

2001116364

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2001116367

Product Development

**2001116368**

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RESEARCH CENTER  
RICHMOND, VIRGINIA

MONTHLY PROGRESS REPORTS

Period Covered

September 1-30, 1985

Date Issued

October 15, 1985

2001116365

**CHARGE NUMBER: 2100**

**PROJECT TITLE: NEW CIGARETTE TECHNOLOGY**

**PROJECT LEADER: W. G. HOUCK, JR.**

**PERIOD COVERED: SEPTEMBER, 1985**

**I. Project DATA**

**Objective:**

To develop an adjustable tar cigarette product.

**Status:**

DATA test market of Concord regular & menthol 85's is in progress.

Completed screening of Merit Select prototypes.

DATA menthol 85 POL extended use test in progress.

In-house, one month extended use test in progress (60 panelists/regular and menthol 85's).

**Plans:**

Product optimization	On-going
Process optimization	On-going
Develop Merit Select Prototypes	4th Qtr., 1985

**II. New Package Designs**

**Objective:**

To develop new and novel package designs.

**Status:**

Completed Mall Testing of Alternate "Slide & Tuck" designs.

Completed modified Cricket designs for New York.

**Plans:**

Make Cricket Packs for New York	October, 1985
Larger scale plastic pack testing	1st Qtr., 1986
Develop new package concepts	On-going

2001116370

September, 1985

## V. Microencapsulation

### **Objective:**

To develop the processing and product encapsulation technology for use in Philip Morris products.

**Status:**

SWRI currently developing more frangible flavor capsules for "last puff freshner" concept.

Initial samples of H<sub>2</sub>O capsules coated on foil innerwrap are being tested for H<sub>2</sub>O time release. Additional samples are being incorporated in cigarette packs for aging studies.

### **Plans:**

- |  |          |
|--|----------|
| Continue in-house development of flavor capsules                               | On-going |
| Develop method to apply H <sub>2</sub> O capsules to foil/<br>cigarettes, etc. | On-going |
| Explore techniques for capsule utility in "last<br>puff freshner" concept      | On-going |

Other:

Nicotine Control: Supercritical CO<sub>2</sub> extraction of Bright tobacco was done by contract with A. Muller of West Germany. The extraction gave 10-50% nicotine reduction and the tobacco was still physically and subjectively similar to the control tobacco. Cigarettes were made and evaluated. A memo will follow.

### Plans:

Work is transferred to other groups.

W. G. Houck  
W. G. Houck, Jr.

~~W. G. Houck, Jr.~~

200116372

Process Development

**200116383**

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2001116366

**TITLE:** Marlboro

**PERIOD COVERED:** February 1985 - September, 1985

**DATE:** September 25, 1985

**RESPONSIBLE PERSON:** J. L. Spruill

**TEAM MEMBER(S):** B. Tierney, J. Swain, A. Palmer, J. Etheridge

**OBJECTIVE:**

Maintenance and monitoring of Marlboro family in conjunction with Marlboro Standardization Group.

**STATUS:**

- Completion of RL/BL Series
- POL's 3347 and 3348 released
- Completion of analytical and subjective evaluations of Marlboro L.S. and K.S. (all factory locations) for five normal production dates and the June Standard Run
- September Standard Run II
  - K.S. and L.S. submitted for CI (all factories)
  - Sample selection for subjective evaluations in progress
  - Blend components submitted for analysis
  - Flavors, packaging materials, cut filler under investigation
- Evaluation of Sheet Materials in Marlboro blend in progress
- Modified RLTC Flavor System samples completed

**PLANS:**

Completion of Status material

/abt

CC:

Team Members

F. L. Daylor

R. M. Ikeda

A. G. Kallianos

H. L. Spielberg

W. Claflin

J. L. Spruill

2001116381

**CHARGE NUMBER: 2105**

**PROJECT TITLE: FILTER DEVELOPMENT**

**PROJECT LEADER: W. A. NICHOLS**

**PERIOD COVERED: SEPTEMBER, 1985**

**I. Filter Development - FML (W. Nichols)**

**Objective:**

To develop the technology to manufacture filters from FML polypropylene tow.

**Status:**

A sample shipment of 4,000 filter rods was sent to Pakistan Tobacco Co., Pakistan for evaluation. If testing is satisfactory the remainder of the order, two million filter rods will be shipped.

Production of an order for 150,000 filter rods for India was initiated. To achieve the circumference specification a new garniture was fabricated.

Sample tow was produced at FML, West Drayton, to determine a correlation between changes in resistance to fibrillation and plugmaker efficiency. During production of the tow, resistance to fibrillation changed as a function of orientation temperature. This sample will be tested in October.

Optimization of tow characteristics continued. A high compression (200 tons) bale was evaluated. Debaling was superior to the normal low compression (15 tons) bales. Acquisition of a 200 ton press for baling is being discussed with FML, West Drayton. Several bales of tow produced on the new fibrillator were tested. Improved yields were found to be due to increased pin density on the fibrillator. The supplier erroneously fabricated fibrillator rolls with 34 pins/inch rather than the specified 25 pins/inch. Evaluations are continuing to determine if this advantageous.

**Plans:**

Improve plugmaker efficiency to 85% November, 1985

Prepare samples for additional market evaluation Continuous

**II. Tobacco Extrusion (R. Thesing)**

**Objective:**

To develop an extruded foam tobacco product meeting Focus Goal One requirements.

**Status:**

The extruded rod was subjectively evaluated versus both the unflavored Marlboro and Marlboro Lights models. It was found to be different from both controls but decidedly better than earlier versions. Flavor work will begin with this model.

200116323

Tobacco Extrusion Cont'd.

Following is a list of analytical data:

	CPB (Extruded)	CO4 (Marlboro)	COZ (Marl. Lts.)
Tar (mg/cigt)	13.4	15.8	10.8
TPM (mg/cigt)	17.4	20.0	12.5
Nic (mg/cigt)	0.74	1.06	0.73
Water (mg/cigt)	3.28	3.11	0.99
PC	7.5	8.8	8.1

Additional flavor models with dual filters were submitted for subjective evaluation. These models contain cellulose acetate (CA) and various tobacco blends in the dual filter. Both cased Marlboro dust and uncased Marlboro dust models are being evaluated.

Plans:

Phase II Production Line

4th Qtr., 1985

Subjective evaluation

Continuous

III. Menthol Application (G. Patron)Objective:

Assist in the introduction of menthol on foil into production.

Status:

Analytical and subjective testing of Marlboro 85mm and menthol control and menthol on foil packed test cigarettes produced in early September were completed. All cigarettes were produced using the new Golden Belt alcohol resistant gold stripe foil. Analytically the tow test cigarettes (made with filters with and without modified pz) and the control cigarette were determined to be almost equal. Subjective results by the Flavor panel showed the three models different, favoring the control cigarette slightly.

Preliminary machinability testing of the mentholated ARCO (with fatty acid lubricant) foil was run at semiworks to produce Marlboro 85mm menthol cigarettes.

PM mentholator machine #1 was shipped to Louisville for installation at the LMCP. Production procedures for the menthol on foil application were written.

Plans:

Initiate training of Louisville personnel

November, 1985

2001116374

**IV. Cambridge Lights 20's**

Objective: To develop a 12 mg tar 85 and 100 mm value entry product in both Regular and Menthol to compete against Doral.

Status: Cigarette specifications are being finalized for this product. The Test Market is scheduled for January 6, 1986 in Denver and Des Moines.

**Plans:**

Ship Completed Salesmen's Samples	November 21, 1985
Production Start-up	December 2, 1985
Test Market	January 6, 1986.

**V. Project Bristol**

Objective: To develop a 85 and 100 mm generic cigarette in both Regular and Menthol.

Status: Several models were produced at the request of N.Y. Marketing for field sampling. Nonmenthol cigarettes were produced at two tar levels, 12 and 16 mg, and in both 85 and 100 mm configurations.

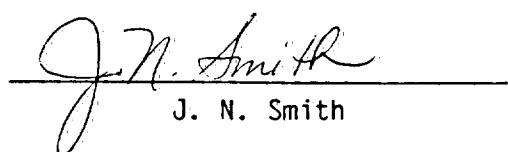
Low cost blends and the foamed binder technology are being investigated for this program.

**VI. Subjective Evaluation of Tipping Paper**

Objective: To determine if there are perceived subjective differences between cigarettes produced with different color tipping papers.

Status: An initial test comparing cigarettes made with cork, cork-on-white and white tipping papers revealed significant differences in perceived harshness. The panelists were blindfolded while smoking and therefore all visual cues were eliminated. Additional testing is planned.

JNS:fs

  
\_\_\_\_\_  
J. N. Smith

200116378

CHARGE NUMBER: 1005  
PROJECT TITLE: Primary Process Development  
PROJECT LEADER: E. G. Craze  
DATE OF REPORT: October 8, 1985

TITLE: HAMBRO DRYER (T. Skidmore)

OBJECTIVE: Evaluate the Hambro vibrating fluidized bed dryer for use in drying tobacco materials during primary processing.

STATUS: Testing is underway to: 1) optimize the operating parameters for Marlboro cut filler, 2) conduct a head-to-head comparison with the Semiworks Adt dryer, and 3) evaluate the Hambro for use in drying strip and stem material.

PLANS: Investigate the potential for using the Hambro as a means for providing second stage reordering of DIET.

TITLE: Tandem Maker Cigarette (D. Albertson)

OBJECTIVE: To produce an ultra low weight cigarette on a continuous basis that has acceptable characteristics.

STATUS: The Tandem Maker hot air drying system was modified to increase the heating capacity and drying performance of the system. Preliminary test results at 200 cpm showed that the drying system was sufficient to produce a 5% OV change. However, firmness and loose ends were unsatisfactory at tobacco weights of approximately 500 mg.

PLANS: An investigation will be conducted to determine why acceptable firmness and loose ends cannot be achieved at target weights of 500-550 mg. of tobacco.

TITLE: Small Scale Processing (T. Skidmore)

OBJECTIVE: Develop a small scale process that will provide cigarette characteristics that are fully comparable to those of the full scale process.

2001116388

**2001116414**

Applied Research

**2001116399**

**TITLE:** Burley Flavor Activity

**PERIOD COVERED:** July - September, 1985

**DATE:** September 25, 1985

**RESPONSIBLE PERSON:** Barbara G. Taylor *B.G.T.*

**TEAM MEMBER(S):** H. Maxwell, V. Willis

**OBJECTIVE**

To develop flavors and/or processes that will contribute to or enhance Burley character in existing and new brands for domestic and international markets.

**STATUS**

**Burley Flavor Development**

A Marlboro-type blend with 0% Burley lamina compensated with Players Navy Cut blend was chosen as the testing medium for Burley flavor application. This blend when tested vs. the Marlboro control in Duo Trio testings, gave the highest Degree of Differences (above 99%) over other tobacco components when evaluated by the Flavor Development panel. However, evaluations by the Factory Panel resulted in NSD. To increase the "n", this blend vs. Marlboro control is presently being evaluated in POL 3383. The close out date is 10/16/85. Once the POL is completed and results are favorable, Burley flavors will be applied to this test blend to reduce the Degree of Difference.

**Non-Volatile Burley Components**

The ethanol/water extractions of C34 Burley have been completed. The 45 fractions obtained have been subjectively evaluated with four fractions warranting further investigation. These fractions have been presented to the Non-Volatile Tobacco Components Activity's RP, Wynn Raymond, for further fractionation.

**PLANS AND TIMETABLE**

**Burley Flavor Development**

POL 3383 - Close Out	10/16/85
Burley Flavor application to test blend	10/16/85
POL Initiated	10/85

**Non-Volatile Burley Components**

Further fractionation of 4 fractions	11/85
Subjective evaluations of sub-fractions	11/85

2001116380

PROJECT NUMBER: 1307

PROJECT TITLE : Reconstituted Tobacco Development

PERIOD COVERED: September 1-30, 1985

PROJECT LEADER: R. G. Uhl

**I. IMPROVED RL SHEET PROPERTIES**

A. Objective

Improve the physical characteristics and blend performance of RL.

B. Status

1. A series of pilot RL test sheets was produced for the subjective evaluation of alternate humectant systems to replace TEG. Sorbitol was found to be subjectively unacceptable. The preferred alternate humectant for RLTC was 4% PG, while a combination of 3% PG/2.5% glycerin was preferred with the 150B flavor system.
2. About 20% of the PG added at the size press is lost in the tunnel dryer. PG losses in the OV test, etc., are being investigated with Analytical Services. Product from proposed Park 500 trials will be evaluated in the Semiworks for pads, survivability and sheet stability after various periods of warehouse storage.

C. Plans

Provide all necessary support for humectant trials at Park 500.

**II. SUBJECTIVE MODIFICATION OF RL**

A. Objective

Improve or modify the subjective character of RL.

B. Status

1. The Votator heat exchangers were examined with a metallurgical consultant. There was no evident corrosion or flaking of the chrome plating on the second exchanger, although scoring exposed the nickel substrate. Scale on the rotor contained phosphorous and calcium. Recommended cleaning agents are being obtained. Samples of acceptable and unacceptable RL/RCB precooked flavors made on this system, as well as current starting materials, are being analyzed offsite for trace metal content via neutron activation and plasma spectroscopy.

2001116390

### III. Project Vanguard

#### Objective:

To develop a consumer acceptable nonburning smoking article.

#### Status:

Electric Cigarette: Basic component testing finished, working on blends and construction variables.

Photoflash: Photoflash bulb testing has been discontinued, we are now negotiating for a stronger power source with GE.

Piezoelectrics: Flavor testing in progress.

#### Plans:

Internal testing (electrically heated)	On-going
Explore new designs	On-going
Optimize current ideas	On-going
Pursue work with GE	On-going

### IV. Dual-Rod Program

#### Objective:

To develop a cigarette product with dual-rod systems for enhanced taste/impact and unique flavor delivery profiles.

#### Status:

Prototype making equipment complete.

Analysis of puff by puff profiles continuing for dual-rod configurations at the ultra low and full flavor tar range.

Subjective evaluations completed on second series. Promising models will be modified with blend and flavor changes for further evaluations.

#### Plans:

Continue prototype design	On-going
Internal testing	4th Qtr., 1985

2001116371

Samples supplied by B. Davies were electrophoresed on a 3-27% SDS polyacrylamine gel. This work was done in conjunction with G. Bokelman to characterize proteins present in extractions of an Acetobacter used to study the synthesis of cellulose.

Jan Hydell

2001116409

CHARGE NUMBER: 1704  
PROJECT TITLE: Supercritical Fluid Processes  
WRITTEN BY: T. M. Howell  
PERIOD COVERED: September, 1985  
DATE OF REPORT: October 8, 1985

## SUPERCritical FLUID SAMPLING

### Objective:

To develop sampling hardware and techniques that will generate accurate and reproducible results from the supercritical fluids laboratory apparatus.

### Status:

Static sampling techniques used with the naphthalene model extraction continued to result in yields 34% to 66% below literature values.

Development Engineering began design procedures for converting the system's magnetically driven mixer to a sample recirculating pump. This conversion makes use of the upper inner portion of the extraction vessel as the pump cavity alleviating the need for a high pressure pump housing.

### Plans:

To abandon static sampling techniques and continue calibrating the system with the model naphthalene extraction when the recirculating pump is in place and functioning properly.

## SCE FACILITY

A proposal to relax the upper temperature limit on the system from 650°F to 450°F was submitted. This reduction is to facilitate the use of polymeric seals within the system. A meeting of the project technical team will be scheduled to discuss any loss in versatility of the system.

2001116394

CHARGE NUMBER: 1503  
PROJECT TITLE: Modified Smoking Materials  
PROJECT LEADER: J. G. Nepomuceno  
PERIOD COVERED: September, 1985  
DATE OF REPORT: October 10, 1985

## I. Tobacco Extrusion

### Objective:

To develop a process for extrusion of foamed tobacco articles.

### Status:

Panel testing of initial prototypes made with a binder system of 1% Klucel, 4% hydroxypropylguar, and 5% starch on the continuous extrusion line found these models to be the best to date. Flavor work to enhance these models is planned and prototypes with additives to reduce mass burn rate and hot collapse are also under evaluation.

### Plans:

Continue efforts to improve subjective response of current model through flavor and blend modifications. Evaluate effects of various additives on mass burn rate and cigarette firmness.

## II. Foamed Filler Binder

### Objective:

To develop a process for applying a subjectively acceptable foamed binder to the tobacco filler in order to improve coal strength, reduce loose ends and allow for weight reduction.

### Status:

Several cigarette models using foamed binder technology were produced with 50, 100 and 200 mg weight reductions. Firmness and loose ends values equivalent to those of the control at standard weight were maintained with as much as 40-50 mg weight reduction.

### Plans:

Further changes to the cigarette construction (filter and paper) are planned in an effort to adjust tar deliveries.

2001116392

CHARGE NUMBER: 0400  
PROJECT TITLE: Tobacco Properties Applications  
PROJECT LEADER: R. S. Mullins  
PERIOD COVERED: September 1985  
DATE OF REPORT: October 10, 1985

## I. Maker Optimization Program

### Objective:

Optimize cigarette makers to reduce degradation of filler and improve product quality.

### Status:

An initial factory test of the carding comb adjustment was conducted in the Manufacturing Center by QA on October 2. No data from the test is available yet.

Since an extended development effort has failed to bring the performance of the belt-feed hopper near a level suitable for use in Manufacturing, no further development or evaluation of the hopper is planned. The modifications are being removed from the hopper and it is being returned to a standard configuration.

### Plans:

Assist in factory evaluation of carding comb adjustment. Continue development and evaluation of the pneumatic picker.

## II. Coal Strength Studies

### Objective:

Investigate the factors affecting cigarette coal strength as measured by the existing coal strength test. Investigate alternate methods of measuring coal strength.

### Status:

A second test has confirmed that the coal failure rate of cigarettes containing dense ends is higher than that of cigarettes without dense ends (52% vs 40%). Tests are planned to determine if this difference is attributable to density differences in the middle portion of the cigarettes.

### Plans:

Continue to conduct tests to determine the factors affecting cigarette coal strength as measured by the existing test. Propose modifications to improve the existing test.

2001116386

### III. Bonded Cigarette Ends

#### Objective:

Determine the effects on cigarette quality of applying binder to the ends of cigarettes. Develop a process for applying binders to the ends of cigarettes at production rates.

#### Status:

Tobacco fallout in the Borgwaldt loose ends test was reduced from 0.6 grams to 0.3 grams by applying 0.02% pectin binder to the ends of the cigarettes. The application of tipping adhesive at a level to 0.05% reduced the fallout to 0.02 grams. At these levels the binders were visually undetectable.

Discussions were held with Dr. Joseph Crowley, a consultant on Ink Jet Printing Technology, regarding the feasibility of using ink jets to apply binders to the ends of cigarettes at production rates. Dr. Crowley felt that this application was within the capabilities of the ink jet technology and recommended that the application be pursued.

#### Plans:

Investigate the effects of alternate binders and application levels on tobacco fallout. Obtain and test an ink jet system for use in this application.

2001116387

2. Two of three precook batches made in the 20 gallon stainless batch reactor were subjectively acceptable (spray application). There were no detectable process or analytical differences between these. The good flavor batches were unacceptable when sized on pilot sheet. Cross products are being made using pilot/Park 500 baseweb/CEL with TC and RL/RCB flavors to isolate the subjective effects of the tobacco substrate from those of the size preparation technique.
3. Precooked flavor has been produced in the Flavor Development 1 liter continuous cooking system. A fully instrumented, multiple vessel continuous system is being designed for installation adjacent to the pilot plant in November.

C. Plans

1. Chemically clean and retest the Votator system.
2. Evaluate pilot/Park 500 cross-product sheets.
3. Review design of the new continuous flavor cooking system.

III. RCB

A. Objective

Develop and evaluate process and product improvements for cast sheets.

B. Status

1. Subjective screening of Modified RCB (TMCI with DAP) handsheets implicates lecithin belt release as a possible source of off notes. Additional tests are being conducted using domestic and European lecithins. Adding RCB quantities of sugar and dry flavors to the formulation gives a definite subjective improvement. Aging the slurry for 30 minutes adds some stemmy character but reduces bitterness and is considered an overall improvement. Aging also improved the physical quality of the handcast sheet.
2. Ammonium tartrate appears to be more effective than tartaric acid in lightening the color of RCB handsheets when the BL Plant makeup sequence is followed. Preliminary screening indicates a subjective limitation of 2% tartrate. Additional testing is in progress.

C. Plans

1. Continue program to improve subjective quality of Modified RCB.
2. Continue evaluation of color-lightening agents for RCB.
3. Provide support for humectant trials at the BL Plant.

/dbb

R. L. Mcl

2001116391

### III. Dry Formed Rods

#### Objective:

To develop a process for producing low density cigarette rods.

#### Status:

Methods of applying binders to the tobacco at the wide tape section of the Hauni Baby were investigated. A long nozzle with multiple orifices for foam application was fabricated and is currently under evaluation. A small ultrasonic nozzle for atomizing the binder solution has been ordered for evaluation.

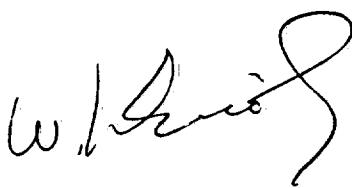
#### Plans:

Continue evaluation of various methods for applying a more complete coverage of binders to the tobacco at the maker.

JGN/lad

2001116393

3. D. Magin, N.B. 8222.
4. J. Wooten
5. W. N. Einolf, "Trip Report: 10th International Mass Spectrometry Conference", Memo to R. H. Cox, September 19, 1985.



2001116422

CHARGE NUMBER : 1801

PROJECT TITLE : Expanded Tobacco Development

PERIOD COVERED: September 1-30, 1985

PROJECT LEADER: G. Gellatly

## I. CO<sub>2</sub> IMPREGNATION

### A. Objective

To find means of CO<sub>2</sub> impregnation of tobacco without clumping.

### B. Status

Two stage spray reordered DIET product in 100% cigarettes of the same firmness did not show any weight difference compared with MC DET. The cigarettes of pilot plant product did, however, show a three fold reduction of loose ends (0.4 gms/50 cigs) compared to MC DET (1.2 gms/50 cigs). Cigarettes containing 30% DIET and MC DET in MF filler did not show this loose end difference. Coal strength analysis is not yet available.

Single stage reordering to 11% on a 20 ft vibrating conveyor showed that this can done without loss of longs and with less CV loss (0.5 units) than in a cylinder (0.8 units). Ordering was done at eight points into free falls from ramps along the conveyor length in 45 seconds (cylinder reordering time is 2 minutes).

A three foot wide vibrating conveyor was installed in the pilot plant to develop design data at up to 4000 lbs/hr tobacco for a commercial installation using tobacco beds of 4" and greater.

### C. Plans

1. Develop design data and OV variability for a commercial design of single and two stage reordering of tobacco beds 4" and greater.
2. Determine the mechanism of CV loss between the tower and reordering.

## II. UNFOLDED STRIP

### A. Objective

To evaluate processing and structural characteristics of unfolded strip for improved filler length and cigarette quality.

**2001116395**

II. ASSISTANCE TO OTHERS

One hundred percent CB mortality was achieved at the 20th St. and Dock St. conditioners used for phytosanitary certification of export blended tobacco (8).

III. REFERENCES

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6. Minor, M. F. Notebook No. 7197, pp. 166-167.
7. Drew, S. Notebook No. 7850, p. 202.
8. Deubler, R. C. Memo to D. T. Wagner. Documentation of Beetle Kill. Sept. 30, 1985.

Daryl J. Faustini

2001116416

**CHARGE NUMBER: 1990**

**PROJECT TITLE: BLEND DEVELOPMENT**

**PROJECT LEADER: C. M. MOOGALIAN**

**PERIOD COVERED: SEPTEMBER, 1985**

**I. Project Tudor**

**Objective:**

Develop a Virginia style cigarette for the U.S. market.

**Status/Plans:**

85's and 100's in the 12mg version were made and shipped to New York for limited testing. However, the testing has been suspended for the time being.

We were recently requested to make 16mg versions (same blend) in both 85's and 100's. These cigarettes will be available to New York for testing in mid-October.

**II. Dunhill Lights**

**Objective:**

To develop a 12mg Winston-type cigarette acceptable to the U.S. market.

**Status/Plans:**

Two HTI tests for this project have been shipped:

1. Dunhill Lts. 85 vs. Marl. Lts. 85  
(HTI 5192) (HTI 1275)
2. Dunhill Lts. 100 vs. Marl. Lts. 100  
(HTI 5193) (HTI 1276)

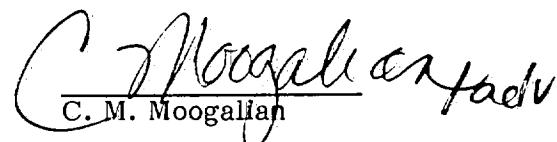
The other tow tests, Dunhill Lts. 85 & 100 vs. Winston 85 & 100, are in analytical and are expected to be shipped by mid-October.

In a Dunhill Lights menthol study, where the regular flavored blend was mentholated at two levels, the higher level (0.69mg smoke menthol) was more acceptable to Marlboro Menthol and Players Menthol smokers, while the lower level (0.53mg smoker menthol) was more acceptable to Merit Menthol smokers. Further menthol studies for this project are in abeyance pending further test market results on Dunhill Lts. Regular.

**Miscellaneous:**

**2001116369**

B&H Menthol with 3% Dark Air-Cured (POL 8046) has been shipped. Close-out date is 10-17-85.

  
C. M. Moogalian

PROJECT CHARGE: 1706

PROJECT TITLE: TOBACCO PHYSICS

PROJECT LEADER: D. B. Losee

PERIOD COVERED: September 1 - 30, 1985

DATE OF REPORT: September 26, 1985

HEAT TRANSFER STUDIES (Waymack, Tiller, Losee)

The required heat removal for extinguishment of a freely smoldering cigarette is greater than the required heat removal for extinguishment of a cigarette in contact with a copper backed fabric. Furthermore, the highest temperature (thermocouple determined) achieved for this cigarette-fabric interface which is backed with copper is only 283°C. The static burn rate for this configuration is approximately 3.3 mm/min compared with the freely smoldering static burn rate of approximately 5 mm/min. This would indicate that the thermal inertia of the substrate has a feedback effect on the free smolder burn rate of cigarettes.

Mass burn rate studies are continuing on a variety of freely smoldering cigarette models. Preliminary indications are that there are not constant differences between calculated mass burn rates which are determined from static burn rate data and these directly determined mass burn rates. The significance of this finding is being pursued.

THERMAL ANALYSIS STUDIES (Tiller)

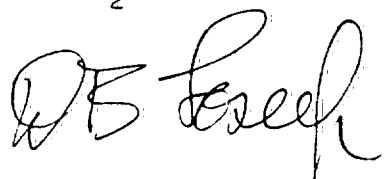
At Peter Martin's request the heat of vaporization of  $\alpha$ -menthol was determined. A value of  $16.1 \pm$  kcal/mole was determined by the Setaram DSC-111 which is in close agreement with a calculated value from vapor pressure-temperature data.

A weight loss study is being conducted on a black fibrous material. This material, a by-product of the laser processing of certain types of printed tipping paper, can reportedly catch on fire.

EXTRACTED TOBACCO STUDIES (Kang)

During tube furnace pyrolysis water extracted tobacco decomposes to produce significantly more selected condensed ring aromatic compounds than an equal weight of supercritical  $\text{CO}_2$  extracted tobacco. This is despite a nearly 50% reduction in tobacco weight after water extraction compared with less than a 10% weight reduction with supercritical  $\text{CO}_2$ .

/ev



2001116402

**2001116435**

**200116438**

Status: Excised segments of leaf mid-vein and aged root growing on Kasperbauer's budding medium<sup>1</sup> did not produce shoots. Transferring mid-vein segments to our standard shoot induction medium has produced normal looking shoots.

Plans: Plantlets will be regenerated and their root chromosomes analyzed for diploidy.

IV. Objective: Produce Coker 319 flue-cured tobacco leaves with lowered nicotine content (Sanders and Weissbecker).

Status: Leaves of grafted Coker 319 tobacco and their controls have been harvested at times designated by the farmer in whose field the plants have been grown. Four primings were cured by Dan Teng in his curing chamber.

Samples have been submitted for chemical analyses and to be made into cigarettes.

Plans: Cigarettes will be smoked by a selected descriptive panel and a report issued as soon as possible.

IV. Miscellaneous (Shulleeta)

Calli established from explants of a homozygous sulfur deficient tobacco mutant (SU/SU) for H. Nakatani have been transferred to shoot inducing medium for plant regeneration for Dr. Nakatani's experiments.

V. References:

1. Kasperbauer, M.J. and Collins, G.B., Reconstitution of diploids from leaf tissue of anther-derived haploids in tobacco. *Crop Sci.*; 12, 998-101 (1972).  
K. Sanders - Notebook No. 8165.  
M. Shulleeta - Notebook No. 8228  
L. Weissbecker - Notebook No. 7998

*L. Weissbecker*  
2001116406

### III. TOBACCO PECTINS (Howard Sun)

As previously reported, tobacco pectin samples have been isolated from Marlboro Turkish, four grades of bright lamina, three grades of burley lamina, three grades of bright stems, and one grade of burley stems. These samples are now being investigated by methanolysis and the methylation analysis procedure to determine their acidic and neutral monosaccharide compositions and linkage patterns.

### IV. MISCELLANEOUS

1. Memo to Dr. J. L. Charles from Gordon H. Bokelman, "Research of Dr. Shuh J. Sheen on Tobacco Proteases and Related Topics," dated September 10, 1985.
2. Memo to Debra Chadick from G. H. Bokelman, "Growth of Acetobacter Xylinum," dated September 13, 1985.
3. Bokelman, Gordon H., W. S. Ryan, Jr., H. H. Sun and G. C. Ruben, "Tobacco Cell Wall Structural Biopolymers," presented at Philip Morris R&D on September 25, 1985.

*Gordon H. Bokelman*

2001116425

CHARGE NUMBER: 6906

-2-

OCTOBER 7, 1985

IV. GLUTATHIONE DEPLETION ASSAY (GDA): EFFECTS OF HYDROGEN PEROXIDE ON GLUTATHIONE LEVELS OF V79 CELLS (2)

An experiment designed to test hydrogen peroxide in a narrow dose range was completed this month. The results from this experiment indicated that there was a dose related depletion of GSH and a dose related increase in GSSG levels.

V. REFERENCE

1. McCoy, William, R. Notebook 8201, pp. 170-175.
2. Deubler, R. Colleen. Notebook 8433, pp. 74-75.

*Charles J. Hackett, Jr.*

2001116432

CHARGE NUMBER: 6904

-2-

OCTOBER 2, 1985

material. As soon as an extraction procedure is operational, the concentrated tobacco material will be assayed for PGR using the Elisa assay developed by IDE-TKE.

REFERENCES

1. Ayers, D. J. 6904 Monthly Progress Report. Monthly Progress Report 85-182; 1985 September 3.
2. Tickle, M. H.; Stagg, D. L. PM Notebook No. 8200 pp. 113-114.
3. Horn, J. L. PM Notebook No. 8229 29-31.
4. Davies, B. D. PM Notebook No. 8005 p. 187.

M. H. Tickle

2001116430

## II. Computer Applications

### Objective:

To write programs in BASIC and provide user assistance for the directorate at O/C.

### Results:

Several changes have been made to DELIVERY, the smoke delivery analysis and data-base program. In particular, a NEWS feature has been added to keep users informed of modifications for error corrections and additional features. Several small bugs have been eliminated and modifications added to enhance the entry of text notes for individual cigarette samples. A new command also allows the user to delete sections of a data storage file from the program rather than using an editor.

## III. Wrapper Modifications

### Objective:

To evaluate modified wrappers for slow burning characteristics.

### Results:

Analysis is in progress of data for repeated measurements of thirteen samples for which both Greiner and Coresta porosity measurements were made. Samples ranged from 3 Greiner seconds to 90 seconds. Preliminary results show that among the several curves examined a LOG-LOG functional form best fits the data. A literature search is in progress to learn what other functional forms have been tried in the past.

## IV. Data Collection Automation

The IBM PC-XT has been received and successfully linked to the Mettler balance. Software is being developed to measure the rate of weight loss of burning cigarettes.

An automated test station has been completed by our Engineering Group to test other cigarette burn properties. Software modifications are in progress for this station to automate data collection.



B. L. Goodman

2001116376

CHARGE NUMBER: Project 1901

PROJECT TITLE: Biochemical Modification of Tobacco

PERIOD COVERED: September 1-30, 1985

PROJECT LEADER: D. M. Teng

DATE OF REPORT: October 7, 1985

### I. Flue Curing Studies

Objective: Alter conventional flue curing condition by maintaining sufficient moisture in the leaf at elevated temperature to enhance the leaf proteases activities so the modification of leaf protein will be more complete.

Status: Total of four sets of experiments were completed. Test curings were conducted at high temperature-high humidity condition for different periods of time. The lamina and stems were then directly dried, using the conditions for normal stem drying.

Plans:

1. Stem the samples
2. Chemical analyses.
3. Biochemical evaluations.
4. Subjective evaluations.

### II. Air Curing Studies

Objectives: To determine the effect of moisture on air-curing of burley tobaccos.

Status: Burley tobacco plants were stalk-cut and hung in chambers, with four moisture levels, for air-curing. Air-curing will continue for the next six to eight weeks.

Plans:

1. Complete the air curing.
2. Sample during different stages of curing.
3. Chemical analyses.
4. Subjective evaluations.

### III. Direct Utilization of Tobacco

Objective: To determine the feasibility of producing acceptable smoking materials with improved chemical, biochemical and subjective characteristics without going through conventional curing.

Status: Control and Ethryl-treated tobaccos were dried and shredded. Stemming, drying and shredding were conducted by the Development personnel.

Plans:

1. Tobacco materials will be treated under different conditions and then submitted for chemical, biochemical and subjective evaluations.

*D. M. Teng*

200116407

CHARGE NUMBER: Project 1720  
PROJECT TITLE: Physicochemical Morphology  
PERIOD COVERED: September 1-30, 1985  
PROJECT LEADER: E. Thomas  
DATE OF REPORT: September 30, 1985

I. Objective: 1. To determine the biochemical and biophysical properties of chloroplast submembrane preparations with respect to oxygen evolution and elucidate the degradation pathways of chloroplast proteins as a function of senescence. (V. Baliga and H. Nakatani)

Status: Intact tobacco chloroplasts were isolated from both ripe and senescent leaves that had been grown under normal conditions in the field. It was determined that the rates of electron transport were ten times less in the chloroplasts isolated from the senescent leaves compared to those from ripe leaves. Chlorophyll (chl) levels were also studied on the ripe and senescent tobacco leaves. While the total chl decreased as the plants matured and senesced, the chl a to chl b ratio remained constant. In another study, chloroplasts isolated from su/su plants were found to have both intact reaction centers and all their associated antenna chl proteins.

Plans: Future studies will continue to document the differences in the photosynthetic electron transport between green and senescent tobacco leaves. Currently, it is planned to use material obtained from coker 319 bright tobacco grown in the Research Center greenhouse. In another study, whole PSII particles will be separated from thylakoid membrane extracts. The oxygen evolving activity from purified components of PSII will be compared to the activity of intact photosynthesising particles.

II. Objective: Study the physical and chemical properties of green tobacco and relate them to the mechanical properties of cured leaf. (E.Taylor, E. Thomas, J. Lyle, P. Echlin)

Status: The study of cellulose microfibrils in green tobacco cell walls by pt replica imaging has been completed and is being issued as a special report. The replicas from this research will also be used in a second study on microfibril organization. The energy dispersive x-ray (EDS) study on element distributions of tobacco cells has continued with over 102 tobacco leaves having been analysed. The data is being statistically studied using the RS/1 software on the VAX. In a separate study, a set of plants spiked with high levels of rubidium and strontium have been examined by EDS. There were detectable levels of both elements in the four major cell types. The only visible sign of stress to these plants was a slight curling of the leaves, which indicated a possible calcium deficiency.

2001116403

STATUS: Testing of the three most promising process variations is continuing. Preliminary data from separate tests indicated that the small scale batch size can be increased from 10 lb. to 20 lb. with no effect on blend uniformity and aftercut application.

PLANS: Complete the evaluation of the three most promising small scale processes.

TITLE: Make/Pack Operations (D. Albertson)

OBJECTIVES: Improve the performance and efficiency of the Semiworks Make/Pack operation.

STATUS: The equipment (Accuray unit and HCF tray filler) for upgrading the MKS maker module has been ordered with delivery expected the first part of 1986. A study is also underway to define the quality assurance requirements for the Semiworks make/pack operation.

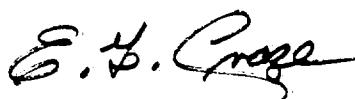
PLANS: Finalize the quality assurance procedures and associated personnel requirements for the Semiworks make/pack operation.

TITLE: MARLBORO STANDARDIZATION (H. Nguyen)

OBJECTIVE: To produce Marlboro cigarettes as specified by the Marlboro Standardization Committee to access the quality of the Semiworks operation.

STATUS: Tobacco samples and process data were obtained at various locations throughout the Semiworks during the second Marlboro Standardization run. The results of sieve and CV/OV data were comparable to those obtained during the first standardization run in June, 1985.

PLANS: The test results are being compiled and will be presented to the Marlboro Standardization Committee.



E. G. Craze

200116389

TITLE: Project Bristol/Players Lights 25's

PERIOD COVERED: July 1, 1985 - October 1, 1985

DATE: October 4, 1985

RESPONSIBLE PERSON: Vivian E. Willis

TEAM MEMBERS: J. Spruill, A. Vulovic, and V. Willis

OBJECTIVES:

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Develop a ten milligram 85mm and a twelve milligram 100mm Players Lights regular and menthol to be sold as a twenty-five soft pack (value entry).

STATUS:

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Test Market for the Players Lights regular has been initiated. The Test Market cities are Des Moines and Denver. A market pickup of the Players Lights regular has been completed. No significant differences were found between the Players Lights regular and other cigarettes that have been picked up from the respective areas in the past. Pack seals on the Players Lights samples remain good. Oven volatiles were comparable to past pickups for the area and time in the field. All storage and stability studies on the flavor system and finished product is complete. The Test Market locations are scheduled to be extended in early October.

Work on the menthol companion has continued. A number of menthol flavors have been evaluated. Through internal testing, the menthol flavor for Players Lights has been chosen. The menthol flavor is 8435-142. Additional inhouse and external testing has been scheduled. Stability and storage studies have been initiated on the Players Lights Menthol with aftercut flavor 8435-142. Flavor formulation has been transmitted. Plans for production start-up have been initiated.

PLANS:

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1. Complete testing of Players Lights regular ripper shorts.
2. Complete stability and storage study on menthol flavors and cigarettes.
3. Test ripper shorts for the Players Lights Menthol.
4. Continue to monitor the Players Lights regular Test Market.
5. Determine optimum flavor application and specifications of the Players Lights regular and menthol at various tar ranges.
6. Production plans
  - a. Manufacturing trial Early October
  - b. Salesman samples October 17, 1985
  - c. Manufacturing start-up October 25, 1985
  - d. National introduction (regular and menthol) January, 1986

REFERENCE:

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Notebook numbers: 8435, 8119

V. E. Willis

2001116382

**CHARGE NUMBER: 4009**

**PROJECT TITLE: DEVELOPMENT SMOKE STUDIES**

**PROJECT LEADER: B. L. GOODMAN**

**PERIOD COVERED: SEPTEMBER, 1985**

I. Project Slow

Objectives:

Develop the technology to reduce or mask objectionable odor of sidestream smoke.

Develop a subjectively acceptable cigarette with reduced sidestream visibility.

Status:

Flavor work, including distinctive flavors, is in progress for the 8.5 mg. tar flavor low model with drastically reduced sidestream. A model of this type with regular aftercut is currently under test with the O/C smoking panel. This test should give further guidance in the flavor work.

The full-flavor model, with the tar level currently at 11.5 mg., will be tested by the M/C smoking panel the week of October 14th.

Two tests of sensory evaluation and aerosol measurements of sidestream smoke were completed this month. In both, the Flavor Development sidestream smoke panel evaluated the sidestream odor of cigarettes in the standard test (7 descriptors, 10 point scale, paired comparison format), followed by aerosol measurements with the TSI Piezobalance.

The first test dealt with extruded rod cigarettes without wrapper and with standard KC 137-1 and Ec 18145 low sidestream MgO wrapper. While the odor test did not show any significant differences between the tested cigarettes, the aerosol measurements of the unwrapped rod gave much higher values. The low sidestream wrapper compared to the standard wrapper gave the expected 40-50% reduction in sidestream aerosol concentration.

In the second test, four cigarettes made with wrappers containing graded levels of MgO at two basis weights were evaluated. The results showed the lowest level of sidestream aerosol concentration for the wrappers with 12% MgO. In the case of the 45 g/m<sup>2</sup> basis weight wrapper the low sidestream aerosol concentration was due to a true decrease in sidestream delivery, while for the 25 g/m<sup>2</sup> wrapper the effect was due to the combination of a smaller decrease in sidestream delivery and a significant decrease in static burn rate. The sensory evaluation showed, surprisingly, higher ratings in harshness for the model with the lowest aerosol concentration, the one made with 45 g/m<sup>2</sup> wrapper.

Both sensory tests point to a high contribution of the wrapper to sidestream smoke odor, higher than the weight ratio wrapper to filler would suggest, and higher than previously thought.

200116375

PROJECT CHARGE: 1702

PROJECT TITLE: FILTRATION PHYSICS

PROJECT LEADER: R. W. Dwyer

WRITTEN BY: K. A. Cox

PERIOD COVERED: September 1 - 30, 1985

DATE OF REPORT: September 26, 1985

LOW DENSITY ROD PROGRAM (Ken Cox)

The role of shred modulus, shred cross section and bed density in determining the stiffness or the bulk modulus of a bed of tobacco is already well understood from simple scaling arguments. To understand the more complicated effect of the randomness of shred orientation and shred length on the modulus, we have carried out extensive calculations of the bulk modulus of infinite networks of shreds in two dimensions. The bulk modulus of different network configurations has been obtained by simulating the response of the networks to compression. We have observed a dramatic increase in the bulk modulus as the randomness of the shred orientations is increased and/or the length of the shreds is increased (at constant mass). For the case of completely random orientations the simulation results have suggested a simple interpretation of the role of shred length in determining the modulus of the bed. This simple interpretation has lead to an expression for the modulus as a function of shred length which agrees quantitatively with the results obtained by simulation. This interpretation, which should be equally valid in three dimensions, is now being used to develop an analogous expression for the bulk modulus of three dimensional beds of tobacco which can be validated by experiment.

Kenneth A. Cox

/ev

2001116400

PROJECT CHARGE: 1703

PROJECT TITLE: Cigarette Making Technology

PROJECT LEADER: A. Robinson

PERIOD COVERED: September 1 - 30, 1985

DATE OF REPORT: September 30, 1985

FLAVORS ADSORPTION STUDIES (A. Robinson, V. P. Henderson)

Contact angle, surface tension test methods and the concepts of critical surface tension have been used to determine the adsorption behavior characteristics of Bright Casing, Burley Casing, Burley Top and Marlboro AC on tobaccos. The investigation was initiated in support of the "Flavorants Spotting Study".

Preliminary results show that the conventional Bright Casing and Marlboro Aftercut do not readily spread onto Oriental tobacco surfaces (i.e., they bead). This observation is consistent with predictions from critical surface tension data.

This investigation will continue with other tobaccos. In addition, the experiments will be expanded to include the measurements as a function of temperature. From the temperature results we hope to gain insight about optimum casings and flavorants application temperatures.

TOBACCO ADHESIVE STUDIES (T. E. Majewski)

Ten percent of calcium acetate based on the weight of sucrose present in an aqueous solution forms a material with desirable adhesive properties. The lower limit of the amount of calcium acetate needed to form an adhesive material from sucrose and be foam stable with licorice is being determined. Additionally, the correct temperature to which the mixture should be heated to prepare the sucrose/calcium acetate mixture is also being studied.

POLYPROPYLENE FILTERS ADHESIVE STUDIES (T. E. Majewski, A. Robinson, V. P. Henderson)

Work continues in characterizing the adhesives used on the polypropylene filter tow. The initial critical surface tension ( $\gamma_c$ ) and surface tension ( $\gamma_{LV}$ ) results suggested that the tow material and the adhesive were compatible (i.e., had a good surface match). However, our more recent results indicate the materials are too compatible. There is a large disparity between the tow  $\gamma_c$  value of 82 dynes/cm and the adhesive  $\gamma_{LV}$  value of 31 dynes/cm. Ideally, for best adhesion  $\gamma_c \leq \gamma_{LV}$ , but when there is such a large difference with  $\gamma_{LV}$  being on the lower side, the liquid is expected to be completely absorbed by the substrate. This behavior was observed experimentally.

/ev

A. Robinson

200116401

**II. REFERENCES**

1. Warfield, A.H., Notebook 8196; Petri, D. Notebook 8006; Hansen, K., Notebook 8215; Yu, T. Notebook 8149. Contributions to this study have been made by all members of 6902, as well as several members of 6908 and 1901.
2. Warfield, A.H., Notebook 8196; Petri, D. Notebook 8006; Hansen, K., Notebook 8215; Yu, T. Notebook 8149.

*A.H.Warfield*

2001116428

CHARGE NUMBER: Project 1904

PROJECT TITLE: Tobacco Physiology and Biochemistry

PERIOD COVERED: September 1-30, 1985

PROJECT LEADER: I.L. Uydess

DATE OF REPORT: October 3, 1985

Objective: To establish the time course and biochemical changes characteristic of tobacco leaves at various stages during senescence.

Status: Two different extractions of greenhouse-grown, Coker 319 green leaf were prepared for use in three biochemical enzyme assays. One set of extractions was made in Pipes buffer, containing KCl, sodium ascorbate, and thiourea, with and without one of two protease inhibitors (Phenyl-Methyl-Sulfonyl-Fluoride, and leupeptin). The second set of extractions was made in Bis-Tris buffer, containing thiourea and KCl, with and without casein and/or leupeptin (a serine and thiol-type protease inhibitor).

Leucine aminopeptidase (LAP), endopeptidase (EP), and carboxypeptidase-A (CP-A) assays were conducted on the aforementioned green leaf extracts. All positive controls using commercially available preparations of each enzyme worked well. LAP was the only enzyme which showed activity in any of these extracts, as well as in previous extracts.

Protein determinations according to the Bradford method were also made on these extracts. It is not clear at this time whether or not the inclusion of PMSF, casein, and/or leupeptin is necessary.

A method for the determination of chlorophyll in tobacco leaves was transferred from Project 1720 to Project 1904. Frozen green leaf, freeze-dried green leaf, and senesced (yellow) leaf were ground with liquid nitrogen and the chlorophyll extracted with 80% acetone. The absorbancy values were measured at 663 nm and 645 nm and corrected against a background absorbancy at 720 nm. As expected, the amount of chlorophyll was found to be greater in the green leaves, than in the yellow, senesced leaves.

Plans: To continue testing for enzyme activities in tobacco extracts prepared from material grown in the greenhouse or in the field under various conditions.

Other Studies:

**2001116408**

Greenhouse grown, Coker 319 leaf material frozen at -70°C in July, 1985 was powdered and lyophilized to determine the dry weight yield from fresh-frozen leaf at different stages of senescence. This material was subsequently submitted to the Analytical Research Division for the purpose of conducting trial determinations of Ca++, starch, nitrogen, and other targeted tobacco components.

In collaboration with the Leaf Department, the Georgia Flue-Cured Tobacco Variety Evaluation Display was attended in Douglas, GA.<sup>4</sup> A total of 372 leaf tobacco samples from the 1985 Regional Farm Test and Regional Small Plot Test were evaluated. There were 14 entries in the Farm Test, and three of these entries were withdrawn due to genetic segregation observed in the field plots. There were 40 entries in Small Plot Test. In evaluating the Farm Test tobaccos, 25% of the displayed material was judged usable by Philip Morris. For the Small Plot Test, 60% was graded as usable material.

The regular greenhouse seeding, transplanting, and production operations were completed for Coker 319, Speight G<sup>4</sup>28, Ky. 10, Ky 14, N. tomentosiformis, N. sylvestris, N. glauca, N. glutinosa, N. longiflora, T.I. 1112, and Oriental Tobaccos.<sup>5</sup>

References:

1. S. Hassam
2. D. Williams
3. G. Chan
4. R. Bass
5. R. Bass, G. West, G. Newell

*Harry* *Guth*

2001116413

CHARGE NUMBER: 4015

**PROJECT TITLE:** Brand Development

PROJECT LEADER: J. N. Smith

PERIOD COVERED: September, 1985

## I. Virginia Slims Lights 120's

Objective: To develop a subjectively acceptable 120 mm version of Virginia Slims Lights Regular and Menthol.

Status: The national introduction of Virginia Slims Lights 120's is scheduled for October 7, 1985.

## II. B&H Lights Deluxe

Objective: To develop a B&H Lights Regular and Menthol with a 24.0 mm circumference for packaging in a deluxe flip top box.

Status: Salesmen's samples and the initial Test Market production were completed in September. Production runs were analytically and subjectively monitored. The Test Market is scheduled for October 7, 1985 in Sacramento and Tulsa/Oklahoma City.

### III. Players Lights 25's

Objective: To develop a 10 mg tar 85 mm and a 12 mg tar 100 mm cigarette in both Regular and Menthol to be sold as a value entry product with 25 cigarettes per pack.

Status: Players Lights Regular 85 and 100 mm is currently in Test Market. This Test Market is scheduled to be expanded on October 7, 1985.

A national introduction of Players Lights 25's in both Regular and Menthol is scheduled for December/January. Salesmen's samples must be shipped by October 21, 1985 and production start-up for the building of inventory for the national introduction begins on October 25, 1985. It is necessary that 1.5 billion cigarettes be produced by the end of 1985.

The flavor system for the menthol companion was selected and specifications were transferred. Internal subjective evaluations of the menthol product are in progress on the OC Panel. POL testing is planned.

Players Lights 85 and 100 mm Regular tested well in recent HTI testing versus Marlboro Lights. This confirms previous internal and POL testing.

## Plans:

## Test Market Expansion of Nonmenthol Players Lights 25's

October 7, 1985

Ship Completed Salesmen's Samples of  
Players Lights 25's Regular and Menthol

October 21, 1985

## Production Start-up for National Introduction

October 25, 1985

## National Introduction

December/January

Source: <https://www.industrydocuments.ucsf.edu/docs/xzfm0000>

PROJECT NUMBER: 6505  
PROJECT TITLE : Special Investigations and Methods Development  
PROJECT LEADER: William R. Harvey  
PERIOD COVERED: September, 1985

#### I. METALS DETERMINATION IN PLANTS

Samples continue to be received for Cd determination. Output has been delayed by instrument downtime. This has been corrected and results should be forthcoming.

#### II. COOKED FLAVOR SUPPORT

##### A. Continuous Flow

Experiments continue in the stainless steel coil. A preliminary experiment indicates laminar flow. Plugging has occurred in the tubing which may have been due to improper flushing. A fast flushing system is now in use.

##### B. Support for Other Processes

IC scans continue to be run in support of this effort.

#### III. CGI SUPPORT

A potentiometric procedure for the determination of chlorides in green tobacco is being developed for use by CGI personnel.

#### IV. RL INVESTIGATIONS

##### A. Sorbitol Determination

A direct determination for sorbitol in RL sheet has been developed. This IC procedure has been written and will be issued shortly.

##### B. Volatile Acids in RL

Work on the above procedure continues. An effort is in progress which might make the TVA procedure correlate better with the total of formic acid and acetic acid determinations by ion chromatography.

#### V. MEMOS

1. William R. Harvey to Dr. Richard Cox, "Stability of glucosylamine and 2,5 deoxyfructosazine", September 3, 1985.
2. W. Harvey to R. Comes, "Chromium and Nickel in Precook Samples 7501-94, -115 and -142", September 16, 1985.

200116426

William R. Harvey

CHARGE NUMBER: 1806

PROJECT TITLE: New Tobacco Processes

PROJECT LEADER: S. R. Wagoner

PERIOD COVERED: September, 1985

## I. WHOLE LEAF CUTTING

### A. Objective

Develop a process for cutting and separating whole leaf into cigarette filler.

### B. Status

In the evaluation to determine the maximum filler length available by hand stemming whole cut leaf filler at the cutter exit, cigarette results showed no advantage for the whole cut leaf filler compared to a strip control. Even though the test filler was longer ( $>6$  mesh = 7.8 vs 4.6%) exit the maker garniture, loose ends results indicated that the test cigarettes produced slightly higher fallout than the control cigarettes (0.61 vs 0.47 g/50 cigt). The compacimetric firmness results were atypical as the plotted lines of firmness vs density intersected at 0.242 g/cc with a firmness of 41.6 mm\*10. At higher densities, the test cigarettes were less firm relative to the control. Due to this, the cigarette analyses will be retested.

Three batches of whole cut leaf, immediately exit the cutter, were superheated in the 8" tower to aid in the subsequent stem separation in the VT separator. Product yield (85%) and lamina recovery (97%) were higher than in normal processing. However, probably due to the loss in moisture through the tower, sieve analyses showed the VT product to be somewhat shorter than normal ( $>6$  mesh = 54 vs 62%).

### C. Plans

Complete the cigarette comparison (firmness, loose ends, ripped CV/OV) evaluating whole cut leaf filler produced by the D&F feeder with the pneumatic stripper.

200116397

## II. STEM TREATMENT

### A. Objective

To develop methods of improving the fibrillation and subjective character of bright and burley stems.

CHARGE NUMBER: 4020

PROJECT TITLE: TOBACCO STUDIES

PROJECT LEADER: H. V. LANZILLOTTI

PERIOD COVERED: SEPTEMBER, 1985

I. Nonvolatile Components of Tobacco

Objective:

To separate, identify and subjectively evaluate nonvolatile components of Bright and Burley tobaccos.

Status:

Equipment is being assembled to enhance the prepscale separation of components from Bright tobacco extract fractions. These fractions were initially separated by column chromatography and have been subjectively determined to enhance the flue cured tobacco character of 100% DIET cigarettes.

II. RL

Objective:

Conduct investigations with Reconstituted Tobacco Development and Cooked Flavor Groups to improve the smoking properties of RL.

RL/RCB Status:

Cigarettes made from 100% unflavored RL treated with isolated RL/RCB precooked flavor components are being subjectively evaluated. Evaluations to date indicate that the two isomers of deoxyfructosazine contribute subjective characteristics which are different for each isomer. In addition, when the two are applied at the ratio found in precooked flavor, they produce yet another set of characteristics. Experiments of this type are needed to identify components important to RL/RCB subjectives and may give direction to efforts to improve the smoking properties of reconstituted tobacco in general.

Experiments are being conducted with a continuous cooked flavor reactor to determine if acceptable flavors can be produced using residence times predicted from theory based on experience with the batch mix reactor. Also, the ratio of reactants is being investigated in these experiments to elucidate reaction chemistry for specifications and to optimize process parameters. Initial results indicate that these parameters will be different from those found for the batch mix.

Reactor equipment is being updated as ordered equipment is received. Results to date warrant proceeding with current experiments without waiting for a more idealized system to be built. Included in the ordered equipment are parts for multiple stage reactors.

**2001116379**

*H. V. Lanzillotti*  
H. V. Lanzillotti

B. Status

To modify the subjective character, several sets of tests were conducted treating washed shredded bright stems with cellulase enzymes. Variations in testing included the type of cellulase, treatment time, and the addition of oriental tobacco after treatment. Preliminary subjective screening indicated that different cellulase enzymes did produce differing responses.

C. Plans

With the aid of Biochemical Research, conduct a study to characterize the enzyme solution that has been producing the most positive results.

III. MECHANISM STUDIES

A. Objective

To determine the mechanisms that are important to the processing of tobacco, such as attrition, drying, fluidization, etc.

B. Status

Testing was conducted in the 5' batch cylinder examining the effect of flight configuration and rpm on filler cascade formation. Visual observation indicated that, at a constant rpm, twelve flights angled downward from the wall produced the most uniform cascade. In general, increasing the rpm from 13 to 19 only increased the trajectory and turbulence of the filler. PM USA Engineering will use this information as a baseline for after-cut flavor application improvement at Cabarrus.

To determine whether the way in which burley spray is prepared has an effect on subjective and/or processing characteristics, a study was undertaken to identify any differences among the processing locations. Several differences, including storage temperature, processing equipment, and order of component addition were found at the MC, Stockton Street, Louisville, and Cabarrus.

C. Plans

Conduct a test to determine whether the burley spray preparation techniques are producing flavors with differing subjective responses.

/dbb

*J. R. Wagoner*

2001116398

and the packaging of this brand is similar to B & H DeLuxe Ultra Lights 100 (Box) cigarettes.

R. J. Reynolds is test marketing a new version of Salem 80 (Box) cigarettes in Pennsylvania. These cigarettes are packaged in a green flip-top box and have cork tipping paper. This cigarette delivers 16 mg tar, 1.1 mg nicotine and 0.5 mg smoke menthol. The word "menthol" does not appear on the package or carton.

Brown & Williamson is test marketing Kool 85 25's in Nashville, Tennessee and Pittsburgh, Pennsylvania. These cigarettes deliver 17 mg tar, 1.2 mg nicotine and 0.5 mg menthol and are similar to Kool 85.

American Brands is test marketing Lucky Strike 100 Menthol cigarettes in Birmingham, Alabama. These cigarettes deliver 10 mg tar, 0.8 mg nicotine and 0.8 mg menthol.

### III. MEMOS, REPORTS AND PRESENTATIONS

- A. Special Report by R. W. Kanipe, Accession No. 85-180, "Menthol Tracking Study," September, 1985.
- B. Memo to C. C. Bright from R. W. Kanipe, "InfraAlyzer Techniques for the Quantitation of Menthol in Aftercut Solutions-Progress Report," September, 17, 1985.
- C. Memo to Joe Nguyen from R. W. Kanipe, "Specific Gravity Determination for Marlboro Casing and Aftercut," September 18, 1985.
- D. Method No. E-39B, "CF in Filler by Gas Chromatography," updated by R. W. Kanipe.
- E. Method No. E58-A, "Propylene Glycol, Glycerine, and Triethylene Glycol in Smoke, Filler, and Sheet Tobacco," updated by R. W. Kanipe.
- F. Presentation by R. W. Kanipe, "Quantitation of Menthol in Aftercut Solutions by InfraAlyzer Techniques," September 23, 1985.

CCB:rad

*C. C. Bright*

2001116437

## II. INSTRUMENTATION UPDATE

The smoke delivery device has been interfaced with the new olfactometer and is currently in operation. A problem with residual odors lingering in the olfactometer airstream was observed, and the necessary modifications have been designed and are presently being fabricated to correct the problem.

The computer database was reconfigured by Cadwell laboratories and now allows for the accurate storage and retrieval of 16 channels of ERP recordings. Steve Peterson of CAD is presently writing additional database routines to aid in the manipulation and quantification of ERP waveforms.

## III. THE EFFECTS OF CO<sub>2</sub> ON CIGARETTE SMOKE FLAVOR

CO<sub>2</sub> discrimination training and testing continues.<sup>3</sup> To date, three subjects have completed training and have been retested. Preliminary data from these subjects indicate that training does facilitate a subject's ability to detect CO<sub>2</sub>. Training has been initiated in three additional subjects, and upon completion, these subjects will also be retested in order to validate these findings.

## IV. REFERENCES

1. Wannamaker, I. Notebook No. 8207, pp. 55-132.
2. Tindall, J. Personal Communication. 1985 September 19.
3. Hayes, C. S. Notebook No. 8150, pp. 104-105, pp. 113-115.

C. L. Hayes

2001116418

PROJECT NUMBER: 1756  
PROJECT TITLE : Analytical Sensory Correlations  
PROJECT LEADER: B. W. Good  
PERIOD COVERED: September 1985

### I. RL vs. RCB SMOKE

#### A. TPM

Seven types of RL/RCB cigarettes were received from Duane Watson (RCB, RLTC, RL150B, and four Park 500 trial samples of RL-RCB) for characterization and comparison by multivariate analysis of TPM data. TPM components are being chromatographically analyzed by the modified fractionation method. Each sample is analyzed four times. The data, which is organized for BMDP analysis, contains intensity measurements of 289 TPM components (93 from the basic fraction, and 98 each from the acidic and neutral fractions. The data collection is near completion.

#### B. Organic Gas Phase

The above cigarettes are also being analyzed for organic gas phase components by both flame ionization and nitrogen-specific detectors. The data, containing peak areas of 57 components and 20 nitrogen-containing compounds, is being organized for BMDP analysis. Each sample is analyzed three times.

### II. ORIENTAL TOBACCO

The analysis of eight volatile fatty acids in filler and TPM were completed for four grades of oriental tobaccos (YUP, TIW, BMU and GKH) and the DBC bright, burley and oriental smoked by the North Complex Smoking Panel. The average values from four replicates of each sample show a relative standard deviation of 5-10% in filler and 5-15% in TPM. The data, reduced to BMDP compatible format, indicated the general trend of the acid contents in filler and TPM to be YUP>TIW>DBC oriental>BMU>GKH. The differential is less in TPM. The TPM of DBC bright contains the highest levels of propionic, isobutyric, butyric and valeric acids in the group. The GKH contains the least amount of B-methylvaleric acid and YUP, the most. This data set, together with abienol and scleral data, will be analyzed by BMDP and correlated with subjective data, when available.

### III. LABSAM

#### A. Online Handler

The on-line handler programs used to collect PE and OV data were modified to store interim information (tare and sample weights) in keyed rather than sequential files. This change speeds program execution and increases data validity. Similar changes are planned for the ash, HWS and solids programs.

#### B. Leaf Department Reporting

Changes were made to the leaf department reporting program to include lot numbers for Richmond by product materials and to enhance output format. Plans include the incorporation of oriental and off shore tobaccos.

2001116423

B good

CHARGE NUMBER: 0307  
PROJECT TITLE: Measurements Development  
PROJECT LEADER: D. R. Fox  
PERIOD COVERED: September, 1985  
DATE OF REPORT: October 8, 1985

## I. Mathematical Modelling

### Objective:

To maintain the tobacco models for filling power and cigarette firmness by incorporating new materials and measurement methods, conducting related studies of physical properties, and providing predictions based on the models.

### Status:

Initial work was completed on a computer spreadsheet version of the cigarette and tobacco modelling system. A preliminary system is now operational on an IBM PC using the Symphony program from Lotus. Blend formulas and independent parameters can be entered into the spreadsheet and tobacco and cigarette properties are calculated.

### Plans:

Additional features will be added to the spreadsheet system to make it easier for the casual computer user to use. When completed, the system will be documented.

## II. Moisture Measurement

### Objective:

To evaluate new or improved methods for the measurement of moisture in filler and cigarettes.

### Status:

Comparison runs were completed with two ovens set up comparably using the new air flow measurement technique. A combination of previously developed baffles from QA and additional modifications was used to result in approximately equal air flows on the top and bottom shelves. Results from the test were promising, as the shelf-to-shelf difference was eliminated. Several runs were required to show that the ovens were not statistically different from one another. At this point, the work has progressed to where the run-to-run variation inherent in the test is significant, requiring multiple runs. Again, all of the significant differences encountered in the test program can be attributed to air flow differences.

**2001116384**

To demonstrate the setup procedures, an oven from the BL plant was obtained that had been taken out of service because of poor performance. Checkout of the oven revealed numerous problems. A caramel-colored liquid was soaking the oven insulation, which was causing large temperature differences within the oven. The liquid was analyzed and found to be composed of BL-type materials. The source of the liquid is being investigated. In addition, air flows were significantly higher than in typical ovens (above 200 fpm on the top shelf compared to 150 fpm). These problems are being corrected, including reinsulation of the oven.

Plans: Four cell types from the leaves from each of the five stages of growth will be analyzed for Na, Mg, P, S, Cl, K, and Ca with the use of the energy dispersive x-ray spectrometer. Further studies on the organization of the cell wall microfibrils will be carried out on frozen hydrated protoplasts using the SEM and Pt replicas using the TEM. The Rb and Sr concentrations in the spiked plants will be compared to the K and Ca levels of normal plants. The distributions of Rb and Sr should respectively mimic the distributions of K and Ca.

SERVICE WORK: The composition of some deposits on a grinder blade was determined at the request of H. Merritt. Assistance was provided to the Entomology Group in a light microscopy study of a nematode. The image analysis system was used to assist process engineering in their studies on shred length as a function rag size. The composition of a unknown slurry from D-Pilot Plant was determined by EDS to be primarily Fe and Cr in dilute sulfuric acid. A section of clutch lining was examined by EDS for the presence of asbestos. No asbestos fibers were present.



2001116404

CHARGE NUMBER: 6906  
PROGRAM TITLE: BIOLOGICAL EFFECTS OF SMOKE  
PERIOD COVERED: SEPTEMBER 1-30, 1985  
PROJECT LEADER: D. J. AYERS  
DATE OF REPORT: OCTOBER 7, 1985  
WRITTEN BY: C. J. HACKETT, JR.

I. SALMONELLA/MICROSOME (S/M) MUTATION ASSAY: TA98 ACTIVITY WITH METABOLIC ACTIVATION (+S9) OF IMPACTION TRAP (IT) CSC COLLECTED FROM WASHED SHREDDED BRIGHT STEMS OR CELLULASE TREATED WASHED SHREDDED BRIGHT STEMS WITH OR WITHOUT FLAVORS ADDED TO THE FILLER (1)

Strain TA98 +S9 was used in the S/M assay to compare the activity of the control cigarettes (IT CSC obtained from the washed shredded bright stem) to the activity of the IT CSC obtained from cellulase treated washed shredded bright stems with or without flavors added to the filler. There was no significant difference in CSC specific activity (S.A.) between the two cellulase treated cigarettes; i.e., the added flavors had no effect on S.A.. Although the cellulase treatment appeared to lower S.A., the control was made from a different batch of bright stems.

II. S/M TA98 +S9 ACTIVITY OF IT CSC COLLECTED FROM LYOPHILIZED GREEN VERSUS CURED BRIGHT TABACCO FILLER (1)

An experiment was conducted using TA98 +S9 to compare the activity of IT CSC collected from lyophilized green versus cured bright tobacco filler. All of the samples were assayed according to the standard procedures. The results indicated that the CSC from the lyophilized bright green (mid stalk) sample was significantly higher (~25%) in S.A. than the CSC from the flue cured bright (lower stalk) sample.

III. S/M TA98 +S9 AND TA100 +S9 DETERMINATION OF S.A. OF IT CSCs COLLECTED FROM CONTROL AND A VARIETY OF DEPROTEINIZED DBC BURLEY HANDMADE CIGARETTES (1)

Strains TA98 +S9 and TA100 +S9 were used in the S/M assay to determine the S.A. of IT CSC obtained from the control and a variety of deproteinized DBC burley handmade cigarettes submitted by Dr. W. Hempfling. There was no major difference in the rank order of the CSC S.A. between strain TA98 and TA100. The pronase treated sample produced the most significant reduction in S.A. as compared to the control sample (TA98 and TA100). The possible correlation of S.A. with protein and/or total nitrogen values are awaiting completion of the analytical data.

2001116431

A new oven, sold by American Scientific and made in Japan, was received for evaluation. The oven is quite large, but includes some interesting features in the control system. Testing is being planned to evaluate the oven.

Plans:

Upon correction of the BL plant oven, samples will be tested for comparison with other ovens. The repair procedure will also be documented so that BL plant personnel can inspect their other ovens.

### III. Materials Evaluation Facility

Objective:

To provide physical testing services (CV, OV, firmness, loose ends, etc.) to groups inside and outside R&D.

Status:

A quotation was received from Honeywell to install a new Direct Digital Control (DDC) system in the lab. This system would permit multiple RH sensors, sensor calibration, and logging of conditions. The quotation is under review.

Upgrades to the original two CV-OV systems in the lab to eliminate obsolete equipment were completed. In addition, the new compacimeters were received from FTR. These new instruments are microprocessor controlled and are much easier to use than the older models.

A number of alternatives were investigated for the new weight selector. Official word was received from Hauni that they have discontinued their instrument. An instrument from SODIM in France was studied but was rejected because of the potential service problems, since SODIM is not represented in the U.S. A third device, from Filtrona, was rejected as not meeting our requirements. As a result, it appears that we will have to construct our own instrument.

Plans:

The Honeywell proposal will be reviewed with the Development and Plant Engineering groups for their input. Program development will begin for a PC-based workstation for the new compacimeters. Paperwork for a new weight selector will be initiated.

200116385

CHARGE NUMBER: 2525  
PROJECT TITLE: NATURAL PRODUCTS CHEMISTRY AND BOTANICAL INVESTIGATIONS  
PROJECT LEADER: H. J. GRUBBS  
PERIOD COVERED: SEPTEMBER 1-30, 1985  
DATE OF REPORT: OCTOBER 10, 1985

In preparation for analysis of  $^{14}\text{C}$ -n-alkanes, preliminary experiments using unlabelled n-alkanes have been carried out. The current HPLC instrumentation for radioanalysis includes a refractive index (RI) detector and a radioflow monitor. For the preliminary experiments, detection limits for RI detection of n-alkanes were determined using Alltech n-alkane mixture ( $n\text{-C}_{28}$  thru  $n\text{-C}_{38}$ ).

Using RI detection, the n-alkanes were detectable (< 10% of full scale) as well resolved peaks when 5 $\mu\text{g}$  per alkane was injected. The intensities of the signals were less than 10% for those observed using FID (flame ionization detector). With RI detection, the level of background noise as well as band broadening associated with  $n\text{-C}_{34}$  and larger alkanes made the detection of these alkanes ( $> 5\mu\text{g}$ ) more difficult. As was observed previously with flame ionization runs, tailing and broadening of these longer chain n-alkanes was also evident. Qualitative comparison of the FID and RI methods of detection shows the former to be more sensitive. Once conditions for the radioanalysis of  $^{14}\text{C}$ -alkanes have been finalized, a single radioanalysis HPLC system will be assembled containing the FID, RI, and radioflow detection capabilities.<sup>1</sup>

Work continues on the development of synthetic methodology for production of serricornin, the cigarette beetle sex pheromone. 2,4-Dimethyl-5-oxoheptanenitrile, a second step key intermediate in the synthesis, has been separated into isomers and the isomers have been subjected to selective reduction using sodium cyanoborohydride. The hydroxynitriles obtained (2 pairs of diasteromers) were in turn converted to serricornin isomers using ethylmagnesium bromide. Through the use of  $^{13}\text{C}$ -NMR with the above intermediates, it has been possible to assign the proper structures to all four diastereomers of the hydroxynitrile intermediate. Unfortunately, this sequence has failed to provide a potential separation technique for the desired natural 4S, 6S, 7S serricornin isomer.<sup>2</sup>

**2001116412**

Exploration of synthetic methodology for the preparation of tobacco glycosides continues. Benzyl 4,6-di-O-acetyl- $\alpha$ -D-mannopyranoside was treated with trimethyl orthobenzoate in DMF to give the corresponding cyclic othobenzoate. Hydrolytic ring opening provided the benzyl 4,6-di-O-acetyl-2-O-benzoyl- $\alpha$ -D-mannopyranoside. Structures were confirmed by NMR. Similarly, methyl 4,6-O-benzylidene- $\alpha$ -D-mannopyranoside was reacted with methyl orthobenzoate in methylene chloride to give the corresponding 2-O-benzoate. The methyl 4,6-O-benzylidene- $\alpha$ -D-mannopyranoside was prepared from methyl mannoside and benzaldehyde. Benzyl, phenethyl, phenylpropyl 2,3,4,6-tetra-O-acetyl- $\beta$ -D-glucopyranosides were prepared from the alcohols and glucose pentaacetate.<sup>3</sup>

CHARGE NUMBER: 6908  
PROGRAM TITLE: SMOKE CONDENSATE STUDIES  
PERIOD COVERED: SEPTEMBER 1 - 30, 1985  
PROJECT LEADER: R. D. KINSER  
DATE OF REPORT: OCTOBER 8, 1985  
WRITTEN BY: S. A. HAUT

### I. NITROSAMINES

It has been observed that the standard deviation of GC/TEA data is greater when smoking experiments are performed using 5 cigarettes compared to 30 cigarettes. The major parameter differences between the two are the purge gas used (air vs. nitrogen), smoke collection time (50 min vs. 20 min), and the number of cigarettes (5 vs. 30). Experiments have been initiated to determine what effects these variables have on the results generated in each instance [1].

Several cigarette blends designed to give reduced TSNA delivery were evaluated. The blends consisted of mixtures of bright, burley, oriental, ES, Virginia sun cured, dark air cured, and low alkaloid tobaccos. The results indicate that those blends containing low alkaloid components yielded lowered filler and mainstream TSNA levels [2]. These results are consistent with those obtained previously for hand-blended cigarettes [3].

Oriental tobacco has been shown to undergo no measurable pyrosynthesis of TSNA during smoking. Oriental X6D4BWA, with TSNA added in amounts equivalent to burley levels, was oversprayed with 3,4-dihydroxyhydrocinnamic acid (one of the polyphenolic compounds found to be active in reducing mainstream TSNA delivery) and smoked this month. One would expect to see no effect on the treated material relative to TSNA-added BWA control if this antioxidant's mode of TSNA inhibition is solely involved with the pyrosynthetic process (and if no alteration in the tobacco's burning characteristics occur). The result of the experiment indicates that there was, in fact, some transfer interference since the levels with added antioxidant are as low as 65% of control. It is possible that the added antioxidant changed the burning character or complexed with the nitrosamines, either destroying them entirely or changing their smoke transfer abilities [4].

Two experiments have been conducted to determine if rosemary oil or solanesol has an effect on MS TSNA levels. Whole rosemary oil was previously shown to have no significant effect on TSNA delivery. Fractions of rosemary oil have been prepared by vacuum distillation and crystallization. If any rosemary oil components are capable of reducing TSNA levels, this activity should be more easily determined in smoking experiments using the more concen-

**2001116433**

PROJECT NUMBER: 1754  
PROJECT TITLE : Spectroscopic Studies of Tobacco and Smoke Components  
PROJECT LEADER: W. N. Einolf  
PERIOD COVERED: September, 1985

NMR (J. Wooten, R. Bassfield, J. Campbell)

$^{13}\text{C}$  NMR was used to follow the appearance and disappearance of peaks in a cooked flavor reaction in a collaborative study with J. Lephardt. By adjusting the concentration of the reactants to control the rate of the reaction, the appearance of products and disappearance of reactants could be followed over a period of three days. Glucosylamine, diglucosylamine, fructose, glucose and fructosazines were monitored.<sup>4</sup>

The collaborative study (with G. Chan) of di-mannose compounds has been completed. Experiments involving elevated temperature and deuterium exchange did not clarify the region of 3.8-4.4 ppm to make possible all proton assignments. However, the two compounds in question (the 2- and 3-substituted compounds reported last month) can be distinguished without these data.<sup>1</sup>

Since requests for two-dimensional NMR analyses have increased, additional experiments have been run on the XL-400 NMR. So far, only one, the carbon-carbon connectivity, does not work on this instrument.<sup>1,4</sup>

MS (D. Magin, W. N. Einolf)

A series of scleral compounds have been submitted for GC/MS evaluation. These include isomers of scleral, methyl, ethyl and isopropyl ethers, sclereolide, anhydroscleral isomers, and scleral phenethyl ether. The data are now being analyzed.<sup>3</sup>

We have received programs from D. Zayelski (USDA, North Dakota) which contain routines he uses to transfer files from one computer to another. We will use these programs in an attempt to transfer files between F. Hsu's data system and our SS-200. Ultimately, we hope to be able to link three data systems - F. Hsu's, our SS-200 and J. Naworal's - so that we can build a data base which will reside in the SS-200 memory.<sup>3</sup>

W. N. Einolf attended and presented a paper at the 10th International Mass Spectrometry Conference. A trip report has been written.<sup>5</sup>

Additional cigarettes were made by D. Petri and are being conditioned. Another control LTF-IIA (new supplier of sodium polypectate and calcium lactate), and LTF-IIA with 10% DAP and 10% arbutin (hydroquinone- $\beta$ -D-glucopyranoside) have been made. The use of internal standards is also being evaluated.<sup>2</sup>

REFERENCES

1. R. Bassfield, N.B. 7398.
2. N. Einolf, N.B. 8040.

200116421

CHARGE NUMBER: 2501  
PROJECT TITLE: SMOKE CHEMISTRY  
PROJECT LEADER: R. H. NEWMAN  
PERIOD COVERED: SEPTEMBER 1-30, 1985  
DATE OF REPORT: OCTOBER 8, 1985

A study of the distribution of radioactivity in the smoke streams from <sup>14</sup>C caffeine added to cigarette filler has now been completed. The following results were obtained.

<u>SMOKE STREAM</u>	<u>% ACTIVITY</u>
SS Gas	13.7
MS Gas	1.2
SS Chamber Wash	14.9
MS Chamber Wash	3.3
SS TPM	30.6
MS TPM	31.8
Butt	4.3
Ash	0.2

The above data is based on tobacco consumed in smoking. An overall average of 98% of the activity was recovered compared to data obtained from filler extracted prior to smoking. Analysis by TLC of the activity on the SS pad and MS pad indicated that better than 95% was the parent <sup>14</sup>C caffeine.

The Hewlett-Packard 5880 gas chromatographs were modified to provide analog output capability to be used in interfacing the H.P.'s with the new lab computer system.

Tomographic reconstructions were performed on a cigarette coal that had been extinguished with N<sub>2</sub>. It appears to be feasible to map to shape of the coal and the relative amount of carbon by reconstructing the cross sections at various positions through the coal.

A computer program is being written to measure the luminance levels in the IR image and to then convert this information to the temperature distributions. This will allow computer analysis of the image with greater flexibility and accuracy without having to rely completely on the limited analysis capabilities of the IR camera itself.

Seven drums of rad waste were collected, packaged according to NRC regulations, and have now been shipped to the disposal site.

200116411

*R.H. Newman*

PROJECT CHARGE: 2106

PROJECT TITLE: APPLIED TECHNOLOGY

PROJECT LEADER: J. L. Banyasz

PERIOD COVERED: September 1 - 30, 1985

DATE OF REPORT: September 26, 1985

PACKAGING (C. D. Owens, T. V. Van Auken)

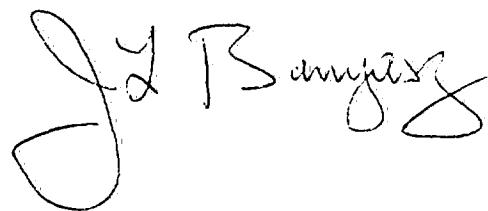
A memo was issued detailing the results with regard to moisture transmission rate versus pack seal efficiency as measured by the IMPS unit. The pack moisture transmission rate was found to fall off exponentially with IMPS value to the permeability limit of the film. Maximum sealing reduced the moisture transmission rate by a factor of about two as compared to a poorly sealed pack. It appears that further reductions would require an improvement in the barrier itself.

A collaborative program with Development was started to evaluate the potential of microencapsulated water as a means of maintaining pack moisture. Samples of three types of microcapsules, received from R. Thiesing, were monitored for rate of water release at RH values of 30, 60 and 90%. The capsules released water at all three RH values though the rate fell off with increasing RH. This work is ongoing..

FLAVOR TRANSFER (A. L. Finley and V. Bowles)

Work continued in the area of defining a reproducible and reversible activation process for silica gels. The procedures previously determined on the DTA were scaled up to the multigram level. Difficulties in the scale-up were encountered due to mass transfer problems which are not a factor with the milligram quantities involved in the DTA experiments. Ongoing experiments indicate that slower heating rates and increased vacuum will probably be required for the larger samples.

/ev



200116410

PROJECT NUMBER: 1758  
PROJECT TITLE: Tobacco Cell Wall Research  
PROJECT LEADER: Gordon H. Bokelman  
PERIOD COVERED: September, 1985

### I. BLEND ANALYSIS (Howard Sun)

A database was designed for analyzing the blend compositions in the cigarettes generated from Tom Skidmore's small scale process development program. The objective of this program is to develop a small scale process for producing cigarettes that is comparable to the full scale process.

In collaboration with Doug Albertson, a series of Marlboro cigarettes produced under different manufacturing conditions and their respective co-generated winnowers are now being examined. A major objective is to determine if the resultant blend composition of the cigarettes varies as a function of the large fluctuations in total winnowers content.

### II. RECONSTITUTED TOBACCO STUDIES (Bill Ryan)

Progress has been made in a study to determine the compositions of cell wall structural biopolymers in commercial samples of RCB, RL and RL baseweb by our standard fractionation procedure (Bokelman, Gordon H., William S. Ryan, Jr. and Elisabeth T. Oakley, "Fractionation of Bright Tobacco," *J. Agric. Food Chem.* 1983, 31, 897-901). Although the calculations cannot be completed until starch determinations are performed by General Analytical, the following results (reported on a percentage dry weight basis) have been obtained.

<u>Component</u>	<u>RCB</u>	<u>RLTC</u>	<u>RL-BW</u>
EtOH Solubles	40.2	37.8	4.6
Aq. Solubles Lost during Dialysis	NA	NA	NA
Acid Detergent Solubles	7.7	4.4	8.3
Pectin	13.2	15.2	27.7
Starch	NA	NA	NA
Protein	6.1	4.6	7.3
Hemicellulose			
Soluble	2.0	3.5	5.8
Associated with Cellulose	1.4	2.2	3.9
Lignin	2.1	2.6	4.0
Cellulose	10.0	15.8	28.3
Ash			
Soluble	2.3	0.9	1.5
Insoluble	6.2	0.8	2.6

NA = Not Available

2001116424

CHARGE NUMBER: Project 1730  
PROJECT TITLE: Plant Tissue Research  
PERIOD COVERED: September 1-30, 1985  
PROJECT LEADER: L. Weissbecker  
DATE OF REPORT: October 3, 1985

I. Objective: Produce tobacco plants with significantly reduced leaf Cd levels (Shulleeta, Spriggs and Weissbecker).

Status: It was determined that Coker 319 protoplasts and protoplast-derived microcallus tolerate Cd to the same extent as suspension grown cultures of Coker 319 haploid cells. A dose response was obtained for protoplasts in the 0-50 ppm Cd range. With protoplasts and protoplast derived microcallus, the effect was influenced by the cell inoculum.

Analytical results have been obtained from Pat Grantham of the Analytical Division on the first set of plantlets exposed to 5 ppm Cd in agar for 2 weeks. Results indicate that as the plant size (weight) increases, the Cd concentration (ppm) decreases. One would expect the Cd concentration vs plant size to remain constant if Cd is passively absorbed from solution. All other agar grown samples are being held in abeyance until this perceived anomaly is resolved.

Of the 9 plantlets analyzed, the ratio of leaf to root Cd concentration varied from 1 to 0.52 with a mean of  $0.73 \pm 0.15$  (1 std. deviation). The high and low Cd containing plantlets had similar plant morphology to each other and to the other plantlets. Results are given below.

Sample	ppm Cd (dry wt.)	gm dry wt.	gm fresh wt.
A1C leaves	449	0.12	3.16
roots	450	0.06	2.09
B5A leaves	195	0.18	3.06
roots	375	0.07	1.67
All Samples leaves	279	0.15	2.62
roots	385	0.06	1.77

2001116405

Plans: Other plantlets are being grown on supports other than agar such as agarose, glass beads and glasswool and their Cd concentration vs plant weight analyzed to determine if correlations exist.

Agar samples within and outside the root mass are also being submitted to Ms. Grantham to determine the ability of Cd to diffuse in agar.

II. Objective: To produce diploid plantlets from haploid Coker 319 plantlets (Shulleeta).

B. Status

Potential advantages and problems associated with strip unfolding in the stemmery were examined. It was shown that drying fresh strip in the all steam atmosphere (8" tower) prevents the curling incurred in the conventional stemmery drying. When unfolded strip was prized in the hogshead to the same weight as DBC bright (850 lbs), it was more loosely packed. Penetration studies showed that it took 20% less force to penetrate unfolded strip hogshead in a horizontal direction than it took for the control. Separating unfolded strip after prizing was not different from control strip.

It was confirmed that recurling of unfolded strip did not recur during casing in a cylinder (no sieve change). It was also confirmed that longer filler is generated from unfolded strip (4.8% +6 mesh) than the control 2.3% +6 mesh) from the cigarette maker. No difference in filler length from unfolded or control strip was observed by cutting on the cross-country feeder/cutter rather than on a normal cutter.

C. Plans

1. Establish where curling of strip takes place in stemmery and primary and determine if this curling can be prevented.
2. Determine the effect of tobacco grade on curling.
3. Establish the feasibility of unfolding strip in non-tower processes.

III. NEW EXPANSION PROCESS DEVELOPMENT

2001116396

A. Objective

To define the fundamental mechanisms governing impregnant retention, puffing, and setting for the development of a new tobacco expansion process with improved product and process attributes.

B. Status

The mixed-gas hydrate (help gas) technology was shown to be effective using tobacco from which the bulk of the "hydrate inhibiting salts" had been extracted. Propane help gas (5 mole %) in 800 psi argon doubled the impregnant retention of argon alone for extracted bright tobacco (8% HWS). The help gas improved product expansion by 10%. Extensive study of hydrate formation phase relationships in the tobacco solubles system will be necessary if we wish to define the role of inhibitor salts on impregnation.

C. Plans

1. Evaluate physical and subjective properties of cigarettes with Reemtsma ET versus PM Berlin DIET.
2. Explore the mechanism of non-thermal setting.

/dbb

J. Goldsmith

CHARGE NUMBER: 1101  
PROGRAM TITLE: ENTOMOLOGICAL RESEARCH  
PERIOD COVERED: SEPTEMBER 1 - 30, 1985  
PROJECT LEADER: D. L. FAUSTINI  
DATE OF REPORT: OCTOBER 4, 1985

## I. CIGARETTE BEETLE PHYSIOLOGICAL STUDIES

### A. Field

Testing has continued in the warehouse evaluating several candidate synthetic pyrethroid space sprays. One particular spray has been effective in killing CB that were contained in cages at 4, 8 and 16 feet. R&D anticipates in making a recommendation for its use by Dec. 1985 (1).

Pheromone traps are still being evaluated as potential monitors of the CB in tobacco warehouses. Since the study was initiated, in June 1985, the pheromone traps have outperformed the suction traps. Results indicate that the beetle population in large warehouses (1.5 mil cu ft) is quite sparse (2).

### B. Laboratory

Cigarette packages exposed to low numbers of adult CB have been evaluated for damage. Infestation levels appear to be slightly lower with the lower exposure level with one exception, that being the non-filter cigarettes. This confirms one of our earlier observations that immediate exposure to a food source in the non-filter package results in greater CB survival and higher infestation (3).

Samples of Mexican tobacco treated with Kabat® were secured, ground and submitted to Universal Leaf for chemical analysis. These samples represent 1/3 of the total amount of tobacco treated with methoprene. If the results are favorable the test will be concluded (4). Ms. Drew has acquired small nail kegs which will be used to simulate "mini-hogsheads" for the evaluation of methoprene treated liners. Candidate liners will include: burlap, paper, and polyethylene materials (5).

Four tobacco molds were evaluated for attraction by the CB in a dual-choice bioassay chamber. The mold Aspergillus niger showed significant attractant properties ( $P<0.001$ ) to adult CB (6).

Several adhesives were evaluated as candidates to trap CB in a R&D designed pheromone trap. Double faced adhesives ranged from vinyl to acrylic to paper. These adhesives will be evaluated for stability at various temperatures, peelability, and trapping efficiency (7).

2001116415

trated fractions. Pure solanesol added to X6D4BVY burley yielded no significant difference from control in MS smoke TSNA levels, suggesting that tobacco waxes may not participate in TSNA formation [4].

## II. PAHs

Work has been completed on the semi-micro column procedure for PAH-III analysis. The first step of this procedure circumvents the problems encountered in activating prep-TLC plates. A small silica gel column is used to prepare PAH-I instead of preparative TLC. Four satisfactory replicates of the method have been obtained [5].

## III. MAINSTREAM SMOKE STUDIES

Cigarettes were handmade from lyophilized green Coker 319 tobacco and cured Coker 319 tobacco. Mainstream smoke was collected from these cigarettes for testing in the Salmonella/ microsome (S/M) assay. Handmade cigarettes were also prepared from six deproteinized burley fillers and a DBC burley control submitted by Dr. W. Hempfling. Weight and RTD selected cigarettes were smoked to yield MS samples for testing in the S/M assay. In a separate experiment, MS was collected from these cigarettes for water analyses [6].

## IV. REFERENCES

1. Morgan, W. R. Notebook No.8218, p. 83-85.
2. Lambert, E. A. Notebook No. 8240, p. 16-18.
3. Tafur, S.; Lambert, E. Tobacco Specific Nitrosamine (TSNA) Analysis of MS Smoke from an Experimental Tobacco Blend Designed to Deliver Adequate Nicotine and Reduced TSNA. Memo to Dr. E. B. Sanders; 1985, April 1.
4. Haut, S. A. Notebook No. 8167, p. 153-155.
5. Levins, R. J. Notebook No. 8109, p. 197-198.
6. Hellams, R. D. Notebook No. 8456, p. 200.



2001116434

CHARGE NUMBER: 1620  
PROGRAM TITLE: ELECTROPHYSIOLOGICAL STUDIES  
PERIOD COVERED: SEPTEMBER 1 - 30, 1985  
PROJECT LEADER: F. P. GULLOTTA  
DATE OF REPORT: SEPTEMBER 30, 1985  
WRITTEN BY: C. S. HAYES

## I. EVENT-RELATED POTENTIALS TO CHEMICAL STIMULI

### A. Nasal Event-Related Potentials (NERPs): Smoke Studies

The first ERPs to cigarette smoke stimuli were recorded utilizing the new olfactometer/smoke delivery system.<sup>1</sup> The responses were simultaneously recorded from 16 electrode sites, permitting a topographical mapping of the smoke ERPs. The stimuli tested were mainstream smoke obtained from uncased DBC Bright and Burley cigarettes.

Preliminary data from three subjects revealed that for both Burley and Bright cigarettes, the largest (i.e., highest amplitude) ERPs were produced ipsilateral to the nostril being stimulated; however, it appeared that Bright ERPs were more localized and of higher amplitude than Burley ERPs. Furthermore, comparisons made between homologous electrode sites demonstrated better inter-hemispheric correlations for Bright ERPs than for Burley. Also, FFT (Fast Fourier Transform) analyses of the data revealed that for Bright ERPs, the relative power appeared to be concentrated between 1.5 and 5.0 Hz; whereas, Burley ERP FFTs appeared to contain more high frequency components. Overall, the data look very promising, suggesting that these measures can be utilized to differentiate between smoke from Bright and Burley cigarettes. If these findings can be confirmed by additional studies, the work will be extended in an attempt to use ERP measures to differentiate among other cigarette types and tobacco blends.

### B. Nasal Event-Related Potentials (NERPs): Difference Testing Employing Cognitive ERPs

Numerical data comparing standard and target ERP waveforms from a single easy target experiment were submitted to John Tindall and Dick Jones of PED for further analyses. Utilizing a multidimensional scaling (MDS) paradigm, it was determined that the ERP waveforms could be described in two dimensions. Additionally, an analysis of variance comparing the ERPs along these dimensions revealed an  $F$  value that was significant at the .008 level!<sup>2</sup> In the future, it may therefore be possible to use MDS measures of ERP waveforms to aid in determining whether, and to what degree, individuals can make flavor discriminations.

**2001116417**

CHARGE NUMBER: 6904  
PROGRAM TITLE: BIOLOGICAL METHODS DEVELOPMENT AND UTILIZATION  
PERIOD COVERED: SEPTEMBER 1 - 30, 1985  
PROJECT LEADER: MIKE PENN  
DATE OF REPORT: OCTOBER 2, 1985  
WRITTEN BY: MIKE TICKLE

#### I. V79 INHIBITION OF METABOLIC COOPERATION (IMC) ASSAY

Previously frozen cell cultures (6-TGs and 6-TGr) were cultured from liquid nitrogen and later evaluated for their response to a solvent and positive control (1). Preliminary results from the IMC assay indicate that the low plating efficiency problem experienced last month was resolved with the subculturing of a lower passage cell line.

Investigations to identify the origin of a fungal contamination were also continued this month (2-3). Several factors were ruled out as being the basis of this contamination. These include the incubators, laminar flow hoods, petri dishes, glassware and trypsin. Even though, preliminary data indicated the medium used in the IMC assay to be contamination free, one contaminated bottle was later found. Because the media was found contaminated, a more intense effort will be taken in the future to evaluate the medium and medium components. However, preliminary results (not yet completely processed) seem to indicate that the fungal contamination has been reduced. No explanation for this change of events can be rationally advanced at this time.

#### II. THIOBARBITURIC ACID (TBA) TEST

A feasibility study evaluating the TBA test was completed (3). Experimental results from this study seem to show that when V79 cells were exposed to whole smoke (trapped by the smoke bubble), an increase in TBA reactive products was seen over the control. The TBA results from whole smoke showed more consistent trends than did CSC.

The continued use of the TBA test is being evaluated, in view of more recent reports of other assays which purportedly have increased sensitivity and specificity for products of lipid peroxidation.

#### III. PLANT GROWTH REGULATOR (PGR) DETERMINATIONS

Collaborative investigations, involving plant growth regulators, were continued this month with Charge Number 1904 (4). Experiments to date have evaluated the potential procedures for extracting IAA and ABA from tobacco

2001116429

CHARGE NUMBER: 8101  
PROGRAM TITLE: Cigarette Testing Services  
SECTION LEADER: Cynthia C. Bright  
PERIOD COVERED: September, 1985

I. TECHNICAL SUPPORT

A. Filter Flare-up (G. Carter)

A test of St. Mortiz cigarettes made with three different tipping papers was performed. These tippings were (a) Ecusta 40436, a zero percent flare-up tipping, (b) Ecusta 40436 with the gold band associated with the St. Moritz brand, and (c) Ton Papier, a French paper, with the gold band. The testing showed that the Ecusta 40436 with the gold band did have a 3% flare-up rate while the Ton Papier with the gold band had zero percent flare-up. The Ton Papier with gold band can be used on the St. Moritz brand.

B. Favor, a Non-Burning, "Cigarette"-shaped Article (C. Bright)

Favor, a non-burning article produced by Advanced Tobacco Products, Inc., has been introduced in San Antonio, Texas. It is designed to be used as a substitute for cigarette smoking and to provide similar nicotine satisfaction to cigarette smoking. There are three versions; Regular, Lights and Menthol, and it has the shape of a cigarette rod.

It is constructed of a hollow plastic (polyethylene terephthalate) tube with cork on white tipping paper at the mouth-end. Inside the tube is a 9 mm plug insert (on the opposite end from the tipping paper) which is polyethylene material impregnated with flavors. The overall length is 84 mm, the circumference is 25 mm, and the total RTD is 90 mm of water.

The major flavor component is nicotine in all three types and the menthol version does contain a significant amount of menthol as well as nicotine. The other flavors are secondary alkaloids which are typical of those found in tobacco extracts. Several other additives were noted, such as, benzyl alcohol, long-chain hydrocarbons, and BHT (a preservative and antioxidant). Each package has warnings printed on it stating that the product "contains nicotine", not to light it, to keep away from children, and not to use if damp or exposed to high temperatures.

II. MARKET ACTIVITY

**2001116436**

Philip Morris has introduced B & H DeLuxe Lights 100 (Box) plain and menthol cigarettes on test market in Sacramento, CA; Tulsa and Oklahoma City, Oklahoma. This cigarette has a 24 mm circumference

CHARGE NUMBER: 6902  
PROGRAM TITLE: BIOCHEMICAL SPECIAL INVESTIGATIONS  
PROJECT LEADER: A. H. WARFIELD  
PERIOD COVERED: SEPTEMBER 1-30, 1985  
DATE OF REPORT: OCTOBER 2, 1985

### I. CURING STUDIES

A. BRIGHT TOBACCO (1): As described in the August summary, this was a time-course study of the nitrate, nitrite, nitrate reductase activity (NRA), nicotine, minor alkaloids, and TSNA levels of Coker 319 tobacco during flue-curing, as well as a study of the effect of vitamin C and vitamin E on the TSNA levels produced. Most of the results from this study have been obtained, although the data have not been fully assessed as yet. The levels of nitrate found in the tobacco were extremely low. No NRA was found, and no nitrite was detected. Levels of TSNA which developed in the tobacco during curing were extremely low, reflecting the low nitrate levels. Further statements concerning the results will be withheld until a more complete analysis of the data can be made.

B. BURLEY TOBACCO (1): Approximately 300 burley plants were cut on 9/17/85 and transported from Appomattox, Va. to R&D on 9/19/85. Zero-time sampling was carried out, and the untreated plants were hung on racks on T-6 to air-cure. Treatment of 15 plants each with 3 separate solutions (or emulsions) containing ascorbyl palmitate, vitamin E acetate, and a mixture of sodium ascorbate and vitamin E acetate was carried out by spraying. In addition a sprayed control, containing only emulsifying agent, was prepared. After drying overnight, these plants were also placed in the racks to cure. Samples of the untreated tobacco are being taken for analysis each week. For treated plants, samples will be taken after one week of curing, and after curing is completed. None of the analytical data is available as yet.

C. V-446 TOBACCO (2): The V-446 tobacco (a nornicotine converter strain) was cut on 9/16/85 after being grown in the greenhouse by Roger Bass et al. (Project 2525). The plants were sampled for NRA as well as chemical analyses, and placed in the curing racks on T-6 on 9/17/85. Further sampling will be carried out weekly until curing is complete. Only a small percentage of the zero-time data has been obtained as of this time. The levels of nornicotine, however, are quite high as expected, even in the zero-time samples. Whether or not the experiment is successful will depend on the levels of nitrate found, since all data obtained thus far in these studies indicate that very low levels of nitrate do not give rise to production of TSNA's.

200116427

PROJECT NUMBER: 1752  
PROJECT TITLE : Optical Spectroscopy of Tobacco and Smoke  
PROJECT LEADER: R. A. Fenner  
PERIOD COVERED: September, 1985

### I. HYDROGEN BONDING STUDY OF PYRAZINE-ETHANOLS

Two series of pyrazine-ethanols being investigated by Y. Houminer as pyrolytic flavor release agents were evaluated for "free" and intramolecularly hydrogen bonded hydroxyl content by infrared transmission spectroscopy. The spectra obtained for the compounds were subjected to a band-fitting analysis to establish the relative proportions of "free" and "bonded" hydroxyl. The results suggest there is an inverse relationship between pyrolysis rates and the proportion of "bonded" hydroxyl when steric effects (of substituents) are the dominate factor controlling intramolecular hydrogen bonding. When electronic as well as steric factors influence the proportion of "bonded" versus "free" hydroxyl, the correlation with pyrolysis rates becomes more difficult to define.

### II. NICOLET 60 SX GC/FT-IR SYSTEM

Problems encountered with system crashes appear to have been eliminated by a complete replacement of the Nicolet computer system. Spurious software handups during GC data collection are still being addressed by programmers at Nicolet. An investigation is currently in progress to evaluate (for Rett Southwick and Dick Howe) products from reactions involving anhydroscclareodiol and related systems by GC/FT-IR.

### III. SIDESTREAM SMOKE ANALYSIS BY TUNABLE DIODE LASER (TDL)

An optical configuration and sampling system have been setup for sidestream smoke analysis. Initial work has focused on water in sidestream. From preliminary studies, the amount of water vapor measured does not appear to depend on the presence of aerosol since sidestream smoke filtered with a Cambridge pad gives the same amount of water vapor (by TDL analysis) as unfiltered smoke. This independence may arise from a cooling of the aerosol (reduced volatilization) as it enters the low pressure, <15 torr, sample cell.

### IV. DETERMINATION OF TOTAL AMMONIA RELEASE DURING CURING

Total ammonia was determined on the curing chamber atmosphere during curing of bright tobacco under normal conditions and under high humidity conditions. Maximum ammonia values were observed during the fourth day of curing for both conditions. There was some difference in the magnitude of ammonia for the two conditions which cannot be explained at this time.

### V. MEMOS

- A. "Preliminary Studies - Ammonia/Menthol", M. E. Parrish to R. H. Cox, September 5, 1985.
- B. "Hydrogen Bonding Study of Pyrazine - Ethanols", R. A. Fenner to Dr. Y. Houminer, September 25, 1985.

2001116420

R.A. Fenner

PROJECT NUMBER: 1740  
PROJECT TITLE : Flavor Components of Tobacco and Filler  
PROJECT LEADER: C. S. Kroustalis  
PERIOD COVERED: September, 1985

## I. FLAVORS

A. Profiling of the volatile pyrazines in the lab scale reactor aging study has continued. After two weeks of storage at ambient and under refrigeration, several components decreased in concentration. These components were: 2-acetyl-5(6)-methylpyrazine, 2-methyl-3-hydroxymethylpyrazine, methyl pyridinone, dimethyl pyridinone, 5(6)-methyl-2-hydroxymethylpyrazine, pyrazinyl ethanol, 5H-cyclopentapyrazine and two M.W. 138 isomers. It should be noted that the methyl hydroxymethylpyrazine isomers, pyrazinyl ethanol and the M.W. 138 isomers increased after two days followed by steep decrease. This aging study will continue for a total of six weeks.

B. A test market Salem Box which contained one third less menthol than Salem Softpack was found to contain 15x isopulegol, 2x menthone, 4x neoisomenthol and 2.4x benzylbenzoate than the Softpack. This indicates that either a different source of menthol is used or these components are added to menthol.

C. Flavor 8206-96 did not contain CF.

D. Kretek cigarettes were analyzed for eugenol. Djarum contained 4.6% and Gudang Garam contained 5.7% eugenol.

## II. OTHERS

A. Approximately 350 RL samples from the RL stabilization program and the Pilot Plant RL humectant replacement study have been analyzed for low molecular acids. Two of the Pilot Plant samples that did not meet subjectives were high in acetic, propionic and beta-methylvaleric acids.

B. Memos

1. C. S. Kroustalis to J. L. Charles, "Analysis of Marlboro and B&H Menthol Cigarettes from Train Wreck", September 24, 1985.
2. C. S. Kroustalis to F. L. Daylor, "1) Salem Box 2) CF in Flavor 8206-96", September 30, 1985.
3. C. S. Kroustalis to R. D. Carpenter, "Marlboro LS-Louisville P#23 (CC #85041)", September 30, 1985.
4. C. S. Kroustalis to R. D. Carpenter, "B&H 100's HP-S/S M#57 P#72 'A' 2-85 (CC #85043)", September 30, 1985.
5. C. S. Kroustalis to R. D. Carpenter, "Marlboro 100's SP-M/C 2A3 'A' 6-85 (CC #85047)", September 30, 1985.

*C. S. Kroustalis*

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